HETEROCYCLES, Vol. 94, No. 6, 2017, pp. 1115 - 1122. © 2017 The Japan Institute of Heterocyclic Chemistry
Received, 12th February, 2017, Accepted, 14th April, 2017, Published online, 21st April, 2017
DOI: 10.3987/COM-17-13673

A GREEN AND HIGHLY EFFICIENT SYNTHESIS OF 5,5-(PHENYLMETHYLENE)BIS(1,3-DIOXANE-4,6-DIONE) DERIVATIVES IN BIOBASED GLUCONIC ACID AQUEOUS SOLUTION

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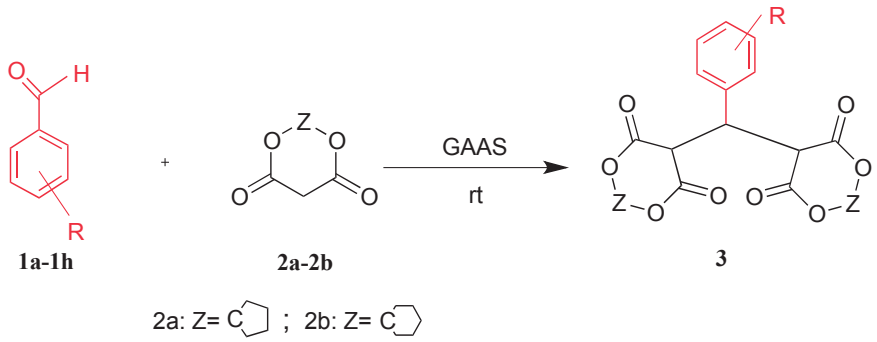
Abstract – A green and highly efficient synthesis of 5,5-(phenylmethylene)bis(1,3-dioxane-4,6-dione) derivatives through tandem Knoevenagel condensation and Michael addition of aromatic aldehydes and 1,3-dioxane-4,6-dione in gluconic acid aqueous solution (GAAS) is described. Gluconic acid aqueous solution could be recycled and reused several times without significant loss of its effciency.

5,5-(Phenylmethylene)bis(1,3-dioxane-4,6-dione) has four carbonyl functionalities along with its tautomeric, that is, keto-enol-forms. Medicinal and organic chemists have shown a great deal of interest in its derivatives owing to their good tyrosinase inhibitory effects and attractive intermediates in the synthesis.[1] In addition, they are also heterocyclic three-ring units in several bioactive such as xanthendione,[2] dispirohydroquinolines,[3] 1,2,4-triazole[1,5-α]pyrimidin-5-ones,[4] and 2,4,10,12-tetraoxadispiro[5.1.5.2]-pentadecane-1,5,9,13-tetraone.[5] Therefore, the preparation of 5,5-(phenylmethylene)bis(1,3-dioxane-4,6-dione) derivatives is of much current importance.

The Knoevenagel condensation and Michael additions of aromatic aldehydes and 1,3-dioxane-4,6-dione have been carried out using a variety of catalysts such as NaOH,[6] KOH,[7] piperidine,[8] Et_3N [9] and proline.[10] However, many of these methods suffer from one or more drawbacks, such as long reaction time, unsatisfactory yields, harsh reaction conditions, and also tedious work-up procedures. The uncatalyzed

reaction has been reported at room temperature in highly polar solvents such as dimethyl sulfoxide (DMSO) and dimethylformamide (DMF), but these solvents are known to be highly toxic, teratogenic, and suspected carcinogens.[11,12] Hence, the introduction of an efficient and eco-friendly methodology in order to confine those mentioned drawbacks is still in great demand.

In order to overcome the above mentioned drawbacks, the development of a clean, safe, and efficient synthetic methodology in green solvents is a focal point of modern organic synthesis. The most commonly used green reaction media are supercritical fluids,[13] ionic liquids,[14] and water.[15] Recently, bio-based solvents such as glycerol,[16] D-xylonic acid,[17] and gluconic acid aqueous solution have increasingly attracted attention. Some organic reactions have been recently examinedin gluconic acid aqueous solution (50 wt%, GAAS). They included Friedel-Crafts alkylations, Michael addition, ring-opening reactions,[18] and multicomponent reactions.[19,20] Herein, we would report gluconic acid aqueous solution as an effective, reusable solvent and catalyst for the synthesis of 5,5-(phenylmethylene)bis(1,3-dioxane-4,6-dione) derivatives via tandem Knoevenagel condensation and Michael additions of aromatic aldehydes and1,3-dioxane-4,6-dione (Scheme 1).



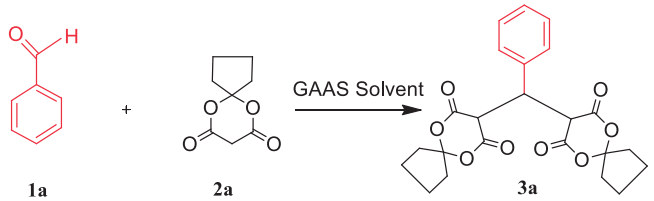
Scheme 1. Synthesis of **3**

For optimizing the reaction conditions, the reaction between benzaldehyde (**1a**) and 2,2-pentylidene-1,3-dioxane-4,6-dione (**2a**) was chosen as a model reaction (Table 1). The reaction is generally catalyzed by basic catalysts, such as NaOH,[6] KOH,[7] piperidine,[8] and Et_3N.[9] A literature survey also stated water [21] has a unique ability to promote this type of reaction. Considering the fact that GAAS is a protic solvent, it would not be unreasonable to expect that GAAS has also similar reaction effects. In order to shed light on the promoting effect of the solvent on the model reaction, we performed the reaction without any catalyst. As shown in Table 1, The yield reached only 36% effectively in neat conditions (Table 1, Entry 1). While the reaction proceeded slightly in non-polar organic solvents, such as toluene and ethyl acetate, the yields sluggishly increased in polar aprotic solvents, such as DMF and DMSO (Table 1, Entries 2-5). A huge improvement was obtained in EtOH, glycerol, water, which are well known polar protic solvents (Table 1, Entries 6-8). To our great delight, a 92% yield was obtained

when gluconic acid aqueous solvent (GAAS) was used as solvent (Table 1, Entry 9). Increasingly, acetic acid and oxalic acid aqueous solution (50%) were examined and displayed less efficiency (Table 1, Entries 10-11). When the amount of gluconic acid aqueous solution was decreased to 3 mL, the product yield only reached 84%, but addition of larger amount of gluconic acid aqueous solution, no further improvement of the product yields was observed (Table 1, Entries 12-13). The optimum reaction time and reaction temperature were also found respectively (Table 1, Entries 14-17). Among the various reaction conditions used in Table 1, it indicated that Entry 9 was the most promising conditions.

Our attention was then turned to the possibility of recycling gluconic acid aqueous solution since the recovery and reuse of the medium are highly preferable for a greener process. After completion of the reaction, the reaction mixture was filtered. The filtrate consisting gluconic acid aqueous solution, was recovered and then subjected to the next run in the model reaction. After four recycles, gluconic acid aqueous solution still showed a high activity and gave the corresponding product in high yield (Table 1, Entry 18).

Table 1. Optimization of reaction conditions for the synthesis of **3a**[a]



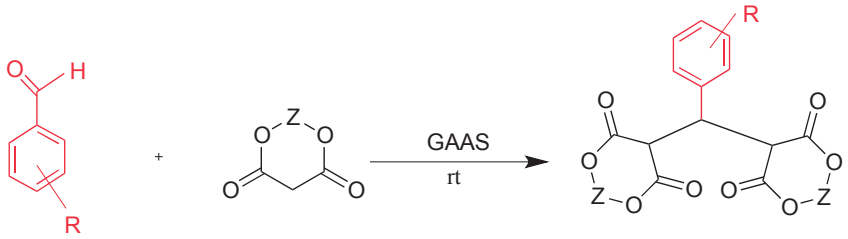
1a **2a** **3a**

Entry	Solvent/mL	Temp.(℃)	Time(h)	Yield (%)[b]
1	none	rt	6	36
2	toluene/4.0	rt	6	44
3	ethyl acetate/4.0	rt	6	60
4	DMF/4.0	rt	6	62
5	DMSO/4.0	rt	6	65
6	EtOH/4.0	rt	6	70
7	glycerol/4.0	rt	6	61
8	H_2O/4.0	rt	5	81
9	GAAS(50%)/4.0	rt	5	92
10	AcOH aqu.(50%)/4.0	rt	5	83
11	oxalic acid aqu.(50%)/4.0	rt	5	80
12	GAAS(50%)/3.0	rt	5	84
13	GAAS(50%)/5.0	rt	5	92
14	GAAS(50%)/4.0	40	5	91
15	GAAS(50%)/4.0	60	5	86
16	GAAS(50%)/4.0	rt	4	90
17	GAAS(50%)/4.0	rt	6	92
18[c]	GAAS(50%)/4.0	rt	5	87

[a]Reaction conditions:benzaldehyde (1 mmol); 2,2-butylidene-1,3-dioxane-4,6-dione (2 mmol); 4.0 mL solvent; [b]Isolated yield; [c]Gluconic acid aqueous solution was reused for the forth time.

With the optimal conditions in hand, various aromatic aldehydes were tested in the presence of 2,2-butylidene-1,3-dioxane-4,6-dione (**2a**) or 2,2-pentylidene-1,3-dioxane-4,6-dione (**2b**). The results were summarized in Table 2. It was found that a wide array of electronically diverse aromatic aldehydes **2a-2h** was tolerant to produce **3a-3n** in good to high yields.

Table 2. Effective synthesis of **3** in GAAS[a]



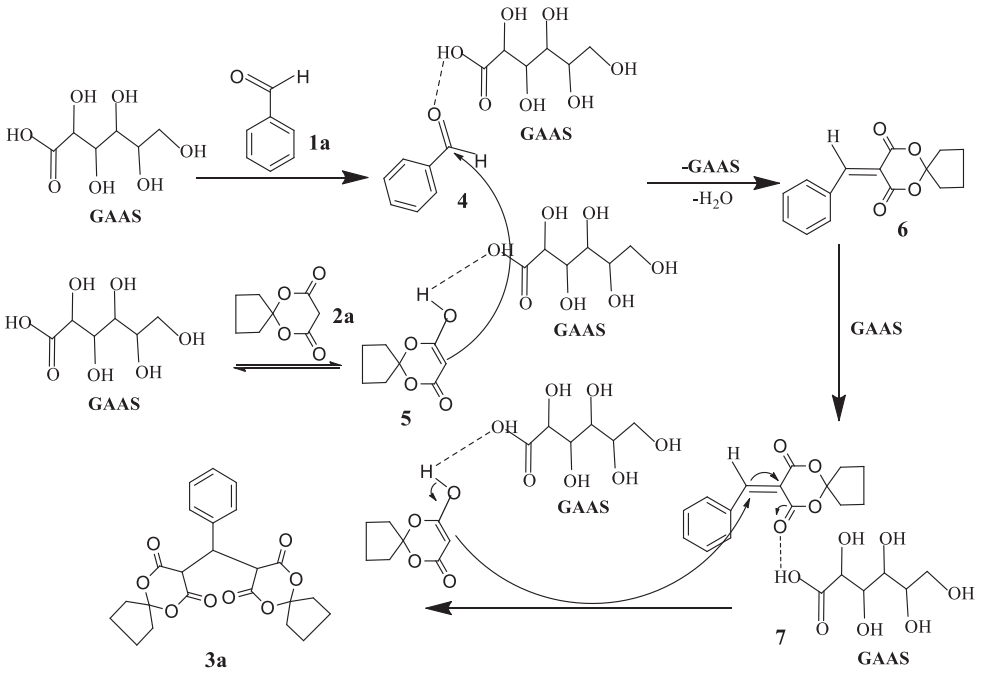
1a-1h **2a-2b** **3**

2a: Z= C ; **2b**: Z= C

Entry	O-Z-O	R	Time(h)	Product	yields (%)[b]
1	2a	**1a**(R=H)	5	**3a**	92
2	2a	**1b**(R=4-F)	4	**3b**	84
3	2a	**1c**(R=4-Cl)	4	**3c**	87
4	2a	**1d**(R=4-Me)	6	**3d**	82
5	2a	**1e**(R=4-NO_2)	3	**3e**	85
6	2a	**1f**(R=4-MeO)	6	**3f**	80
7	2a	**1g**(R=3-Cl)	4	**3g**	93
8	2a	**1h**(R=2-NO_2)	4	**3h**	87
9	2b	**1a**(R=H)	5	**3i**	90
10	2b	**1b**(R=4-F)	4	**3j**	86
11	2b	**1c**(R=4-Cl)	4	**3k**	88
12	2b	**1d**(R=4-Me)	6	**3l**	80
13	2b	**1e**(R=4-NO_2)	3	**3m**	81
14	2b	**1g**(R=3-Cl)	5	**3n**	91

[a] Reaction conditions: aromatic aldehydes (**1a-1h**, 1 mmol); 1,3-dioxane-4,6-diones (2 mmol); GAAS:4.0 mL; temperature rt. [b] Isolated yield.

In accordance with reports from the literature,[21,22] a plausible mechanism for tandem Knoevenagel condensation and Michael additions synthesis of 5,5-(phenylmethylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) **3a** is depicted in Scheme 2. GAAS as a solvent helps in the enoliztion of **2a** by making hydrogen bongds with the –CO_2H of **2a** and, thus it increases the nucleophilic character of the methylene carbon of **2a**. Meanwhile, it also increases the electrophilic character of the carbonyl of **1a** by forming hydrogen bonds with the carbonyl oxygen of benzaldehyde **1a**. After the Knoevenagel condensation reaction, the intermediate **6** is afforded. By the nucleophilic attacking of **5**, the intermediate **7** is transformed to the product **3a**.



Scheme 2. Proposed mechanism for the synthesis of **3a**

EXPERIMENTAL

All chemicals were purchased from Aladdin, Aldrich and Fluka Chemical Companies and without further purification. Melting points were measured on XT-4 digital micro melting point apparatus and are uncorrected. ^{1}H NMR spectra were recorded on a BRUKER AVANCE 400 MHz spectrometer using $CDCl_3$ as the solvent and TMS as the internal standard.^{13}C NMR data were collected on a BRUKER AVANCE 100 MHz instrument with $CDCl_3$ as the solvent and TMS as the internal standard. The analytical MS of the compounds was performed on Agilent LC-MSD Trap VL Apparatus.

General procedure of the preparation of products 3

5,5-(Phenylmethylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3a): To a 25 mL tube equiped with a stirring bar were added 2,2-butylidene-1,3-dioxane-4,6-dione (**2a**, 2 mmol), aromatic aldehyde (**1a**, 1 mmol) and gluconic acid aqueous solution (4.0 mL). The vessel was then sealed with a screw cap and at room temperature for 5.0 h. Upon completion of the reaction, as confirmed by thin-layer chromatography (petroleum ether/EtOAC 4:1), the reaction mixture was filtered. The filtrate consisting gluconic acid aqueous solution, was recovered and then subjected to the next run in the model reaction. The residue was the crude solid product, then washed with water and purified by recrytallization from absolute EtOH to afford the pure product **3a**: White solid; mp 156-158 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.77-1.84 (m, 4 H), 1.86-1.93 (m, 4 H), 2.17-2.30 (m, 8 H), 4.60-4.68 (m, 3 H), 7.28-7.36 (m, 3 H), 7.56 (d, J = 8.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.62, 24.46, 38.17, 39.08, 39.15, 50.48, 114.50, 127.91, 128.58, 129.01, 140.66, 164.53, 165.59; ESI-MS m/z: 429.2 $[M+H]^+$.

5,5-((4-Flurophenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3b): White solid; mp 141-143 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.78-1.85 (m, 4 H), 1.87-1.94 (m, 4 H), 2.17-2.31 (m, 8 H),

4.56-4.70 (m, 3 H), 7.00-7.04 (m, 2 H), 7.53-7.57 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.61, 24.49, 38.16, 38.46, 39.14, 50.51, 114.58, 115.69, 115.90, 130.53, 130.61, 136.16, 164.50, 165.76; ESI-MS *m*/*z*: 466.1$[M+H]^+$.

5,5-((4-Chlorophenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3c): White solid; mp 143-145 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.78-1.85 (m, 4 H), 1.87-1.94 (m, 4 H), 2.16-2.30 (m, 8 H), 4.54-4.70 (m, 3 H), 7.30 (d, *J* = 8.4 Hz, 2 H), 7.50 (d, *J* = 8.4 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.59, 24.48, 38.16, 38.63, 39.13, 50.33, 114.61, 130.24, 130.84, 138.87, 164.44, 165.66; ESI-MS *m*/*z*: 463.1$[M+H]^+$.

5,5-((4-Methylphenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3d): White solid; mp 136-138 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.77-1.84 (m, 4 H), 1.85-1.91 (m, 4 H), 2.15-2.28 (m, 8 H), 2.32 (s, 3 H), 4.55-4.68 (m, 3 H), 7.14 (d, *J* = 8.0 Hz, 2 H), 7.43 (d, *J* = 8.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.07, 22.61, 24.45, 38.14, 38.70, 39.14, 50.56, 114.43, 128.46, 129.63, 137.60, 137.63, 164.58, 165.92; ESI-MS *m*/*z*: 443.2$[M+H]^+$.

5,5-((4-Nitrophenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3e): White solid; mp 134-135 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.80-1.87 (m, 4 H), 1.89-1.96 (m, 4 H), 2.18-2.32 (m, 8 H), 4.66-4.79 (m, 3 H), 7.77 (d, *J* = 8.8 Hz, 2 H), 8.17(d, *J* = 8.8 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.57, 24.49, 38.16, 39.08, 39.14, 49.92, 114.85, 123.99, 130.07, 147.37, 147.41, 164.31, 165.35; ESI-MS *m*/*z*: 474.1$[M+H]^+$.

5,5-((4-Methoxylphenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3f): Light yellow solid; mp 140-142 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.77-1.84 (m, 4 H), 1.86-1.93 (m, 4 H), 2.17-2.30 (m, 8 H), 4.53-4.70 (m, 3 H), 3.78 (s, 3 H), 6.85 (d, *J* = 8.8 Hz, 2 H), 7.61 (d, *J* = 8.8 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.61, 24.44, 38.14, 38.40, 39.15, 50.65, 55.24, 114.21, 114.43, 129.86, 132.46, 137.50, 159.05, 164.59, 165.90; ESI-MS *m*/*z*: 459.2$[M+H]^+$.

5,5-((3-Chlorophenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3g): White solid; mp 144-146 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.79-1.86 (m, 4 H), 1.88-1.95 (m, 4 H), 2.17-2.31 (m, 8 H), 4.57-4.60 (m, 3 H), 7.25-7.31 (m, 2 H), 7.45-7.49 (m, 2 H), 7.57 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.60, 24.49, 38.20, 38.85, 39.14, 50.30, 114.64, 127.20, 128.21, 128.60, 130.22, 134.74, 164.37, 165.70; ESI-MS *m*/*z*: 463.1$[M+H]^+$.

5,5-((2-Nitrophenyl)methylene)bis(2,2-butylidene-1,3-dioxane-4,6-dione) (3h): Light yellow solid; mp 158-160 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.80-1.87 (m, 4 H), 1.89-1.94 (m, 4 H), 2.20-2.28 (m, 8 H), 4.52-5.23 (m, 3 H), 7.45 (t, *J* = 7.2 Hz, 1 H), 7.61 (t, *J* = 7.6 Hz, 1 H), 7.78 (t, *J* = 7.6 Hz, 1 H), 7.45 (t, *J*

= 7.6 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.59, 24.45, 33.24, 38.19, 39.13, 50.26, 114.56, 124.95, 128.60, 128.69, 133.16, 135.29, 150.12, 163.75, 165.66; ESI-MS *m*/*z*: 474.1[M＋H]$^+$.

5,5-(Phenylmethylene)bis(2,2-pentylidene-1,3-dioxane-4,6-dione) (3i): White solid; mp 140-142 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.45-1.50 (m, 4 H), 1.64-1.75 (m, 8 H), 1.85-1.88 (m, 4 H), 1.96-2.07 (m, 4 H), 4.60-4.67 (m, 3 H), 7.29 (d, *J* = 7.2 Hz, 1 H), 7.35 (t, *J* = 7.2 Hz, *J* = 8.0 Hz, 2 H), 7.53 (d, *J* = 7.2 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.85, 22.59, 24.08, 35.54, 37.13, 40.14, 49.42, 106.34, 127.79, 128.41, 128.97, 140.80, 164.02, 165.30; ESI-MS *m*/*z*: 457.2 [M＋H]$^+$.

5,5-((4-Flurophenyl)methylene)bis(2,2-pentylidene-1,3-dioxane-4,6-dione) (3j): White solid; mp 106-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.48-1.51 (m, 4 H), 1.65-1.77 (m, 8 H), 1.89-1.92 (m, 4 H), 1.96-2.07 (m, 4 H), 4.58-4.65 (m, 3 H), 7.01-7.06 (m, 2 H), 7.51-7.55 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.83, 22.61, 24.06, 35.53, 37.07, 39.47, 49.48, 106.45, 115.68, 115.90, 130.30, 130.38, 136.44, 163.99, 165.59; ESI-MS *m*/*z*: 475.2[M＋H]$^+$.

5,5-((4-Chlorophenyl)methylene)bis(2,2-pentylidene-1,3-dioxane-4,6-dione) (3k): White solid; mp 144-146 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47-1.54 (m, 4 H), 1.66-1.77 (m, 8 H), 1.89-1.92 (m, 4 H), 1.97-2.05 (m, 4 H), 4.58-4.63 (m, 3 H), 7.31 (d, *J* = 8.0 Hz, 2 H), 7.49 (d, *J* = 8.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.83, 22.62, 24.06, 35.53, 37.05, 39.60, 49.30, 106.48, 129.08, 130.02, 133.70, 139.24, 163.94, 165.12; ESI-MS *m*/*z*: 491.1[M＋H]$^+$.

5,5-((4-Methylphenyl)methylene)bis(2,2-pentylidene-1,3-dioxane-4,6-dione) (3l): White solid; mp 108-109 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.45-1.50 (m, 4 H), 1.65-1.75 (m, 8 H), 1.86-1.89 (m, 4 H), 1.95-2.05 (m, 4 H), 2.32 (m, 3 H), 4.56-4.65 (m, 3 H), 7.15 (d, *J* = 8.0 Hz, 2 H), 7.42 (d, *J* = 8.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.08, 21.86, 22.59, 24.09, 35.52, 37.12, 39.75, 49.51, 106.29, 128.29, 129.63, 137.52, 137.80, 164.09, 165.34; ESI-MS *m*/*z*: 471.2[M＋H]$^+$.

5,5-((4-Nitrophenyl)methylene)bis(2,2-pentylidene-1,3-dioxane-4,6-dione) (3m): White solid; mp 151-153 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47-1.53 (m, 4 H), 1.66-1.78 (m, 8 H), 1.93-1.96 (m, 4 H), 2.01-2.04 (m, 4 H), 4.70-4.73 (m, 3 H), 7.75 (d, *J* = 8.4 Hz, 2 H), 8.20 (d, *J* = 8.4 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.79, 22.63, 24.02, 35.59, 36.95, 40.04, 48.93, 106.76, 124.10, 129.77, 147.33, 147.92, 163.77, 164.82; ESI-MS *m*/*z*: 502.2[M＋H]$^+$.

5,5-((3-Chlorophenyl)methylene)bis(2,2-pentylidene-1,3-dioxane-4,6-dione) (3n): White solid; mp 146-148 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.47-1.52 (m, 4 H), 1.67-1.76 (m, 8 H), 1.90-1.93 (m, 4 H), 2.01-2.03 (m, 4 H), 4.55-4.60 (m, 3 H), 7.27-7.29 (m, 2 H), 7.43-7.45 (m, 2 H), 7.56 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.83, 22.62, 24.06, 35.56, 37.06, 39.81, 49.25, 106.50, 126.97, 128.06, 128.47, 130.19, 134.72, 143.01, 163.85, 165.11; ESI-MS *m*/*z*: 491.1[M＋H]$^+$.

ACKNOWLEDGEMENTS

This work was supported by the National Natural Science Foundation of China (No. 20566004). We also thank the support from the Graduate Innovation Fundation of Jiangxi Province (No. YC2015-B023).

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