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# Chemoselective Synthesis of Tetraketones in Water Catalyzed by Nanostructured Diphosphate $\text{Na}_2\text{CaP}_2\text{O}_7$

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Green chemistry has now attained the status of a major scientific discipline<sup>1</sup> and studies in this area have led to the development of cleaner and more benign chemical processes, with many new technologies being developed each year. Much effort has been devoted to the use of non-traditional solvents for chemical synthesis. In addition to solvent-free media,<sup>2–3</sup> these unconventional media include water,<sup>4</sup> supercritical  $\text{CO}_2$ ,<sup>5</sup> perfluorinated solvents<sup>6</sup> and ionic liquids.<sup>7–9</sup> The use of water as a medium for organic reactions is one of the latest challenges for modern organic chemists. It will be a major step forward to carry out organic reactions in water for environmental and economic reasons. Furthermore, because of its high polarity, high surface tension, high specific heat capacity and network of hydrogen bonds, water plays a significant role in many reactions.<sup>10–13</sup>

Another aspect of green chemistry is the development of reusable and heterogeneous catalysts under environmentally friendly conditions. From the viewpoint of green chemistry, good recovery and reusability of the catalyst are highly preferable. These concepts are at the center of chemical activity, and research on high selectivity is the driving force for the conception of all new catalytic processes. At present, it is well known that a heterogeneous catalyst must have three characteristics: high activity, selectivity and stability. Nanotechnologies constitute an invaluable tool in catalysis. Considerable progress has been made, but many challenges that deal with the control of the localization of active sites still exist.<sup>14–17</sup>

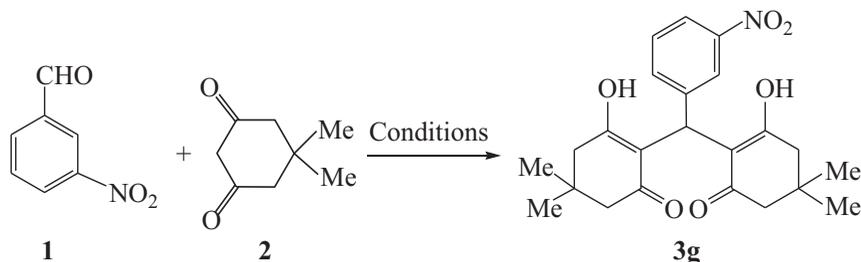
Arylmethylene *bis*-(3-hydroxy-2-cyclohexene-1-one) derivatives (tetraketones) are important substrates used as precursors in the syntheses of xanthenes and acridinediones for laser dye technology.<sup>18</sup> These compounds have also shown potent activity as antioxidants, lipoxigenase inhibitors,<sup>19</sup> and a new clinical class of tyrosinase inhibitors against important dermatological disorders including hyper-pigmentation and skin melanoma.<sup>20</sup>

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**Table 1**  
Optimization of reaction conditions for synthesis of **3 g**



Entry	Catalyst (mol%)	Conditions	Time (min)	Yield (%)
1	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (20)	Water/rt	140	82
2	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (20)	Water/Reflux	5	95
3	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (20)	Water/90 <sup>0</sup> C	60	85
4	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (20)	Water/80 <sup>0</sup> C	90	85
5	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (15)	Water/Reflux	10	85
6	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (25)	Water/Reflux	5	88
7	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (20)	Ethanol/Reflux	20	90
8	Na <sub>2</sub> CaP <sub>2</sub> O <sub>7</sub> (20)	Methanol/Reflux	20	86
9	— <sup>a</sup>	Water/Reflux	180	72

<sup>a</sup>This reaction was carried out in the absence of any catalyst.

aldehydes with electron withdrawing and electron-donating groups as well as heteroaromatic and aliphatic aldehydes were employed. The nature of the substituents on the aromatic ring showed no obvious effect on this conversion; all products were obtained in high yields in relatively short reaction times.

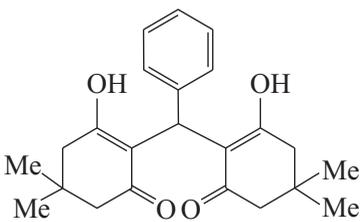
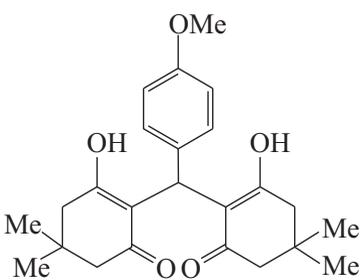
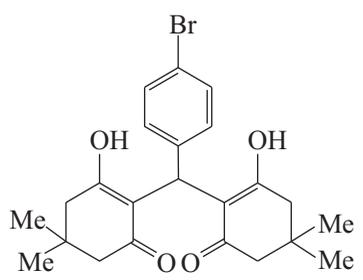
Synthetically, chemoselectivity in chemistry is regarded as one of the most important aspects of organic reactions. In all cases, the desired products (**3 a-r**) were obtained in high yields as the major products. For example, none of the corresponding xanthenes was formed on heating 2,2'-[(3-nitrophenyl)-methylene] bis-(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one) (**3 g**, 1 mmol) in the presence of nanostructured diphosphate Na<sub>2</sub>CaP<sub>2</sub>O<sub>7</sub> (20 mol%) in refluxing water (1 ml); only starting material was recovered.

The scope of the methodology was studied in the preparation of **7 a** and **8 a** by using 1,3-cyclopentanedione (**5**) or 1,3-indanedione (**6**) as one of the components (*Scheme 2*).

In order to illustrate the efficacy of our procedure, results for the preparation of 2,2'-arylmethylene bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) derivatives reported previously are compared with our data (*Table 3*). Our process using nanostructured diphosphate Na<sub>2</sub>CaP<sub>2</sub>O<sub>7</sub> offers several advantages such as excellent yields, a simple procedure, short reaction times, facile work-up and recyclability.

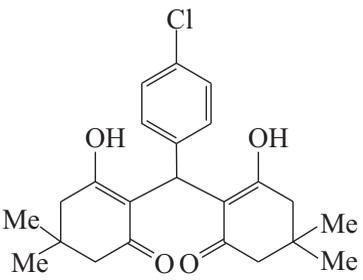
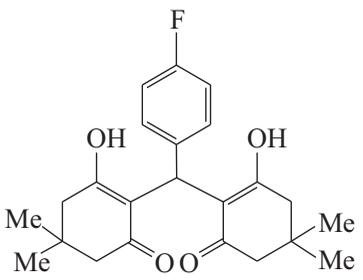
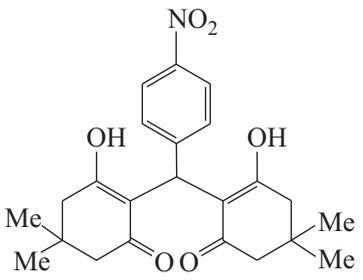
We believe that the surface of Na<sub>2</sub>CaP<sub>2</sub>O<sub>7</sub> presents multiple catalytic active sites. The basic sites (oxygens of P<sub>2</sub>O<sub>7</sub> group and CaO<sub>6</sub> octahedra) may abstract the proton from the dimedone or 1,3-cyclohexanedione. The acidic sites (phosphorus of the P<sub>2</sub>O<sub>7</sub> group, Na<sup>+</sup> and Ca<sup>2+</sup> cations) probably induce the polarization of the C=O bond and facilitate domino Knoevenagel condensation/Michael addition reaction leading to the tetraketones.

**Table 2**  
Synthesis of tetraketone derivatives

Entry	Products (3 a-r)	Yield (%)	Time (min)	Mp (°C)	
				Found	Lit.
1	 <p><b>3a</b></p>	93	10	188–190	186–188 <sup>27</sup>
2	 <p><b>3b</b></p>	92	10	144–146	146–148 <sup>27</sup>
3	 <p><b>3c</b></p>	93	10	157–159	158–159 <sup>28</sup>

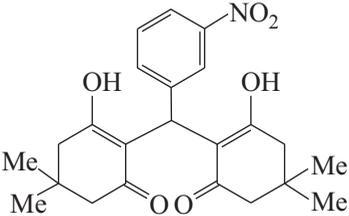
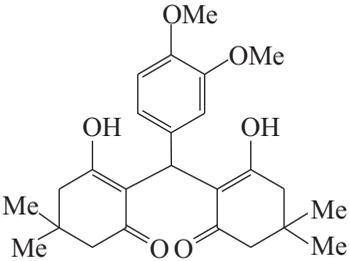
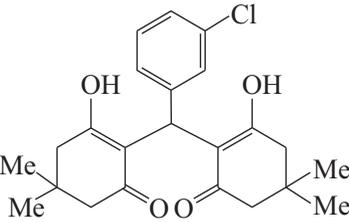
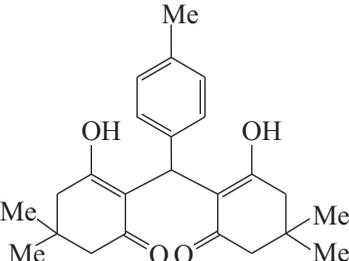
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**Table 2**  
Synthesis of tetraketone derivatives (*Continued*)

Entry	Products (3 a-r)	Yield (%)	Time (min)	Mp ( $^{\circ}\text{C}$ )	
				Found	Lit.
4	 <p><b>3d</b></p>	90	10	143–145	142–144 <sup>27</sup>
5	 <p><b>3e</b></p>	90	10	166–168	167–168 <sup>28</sup>
6	 <p><b>3f</b></p>	94	5	190–192	194–196 <sup>21</sup>

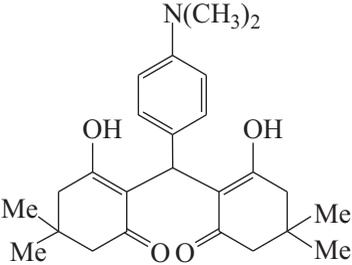
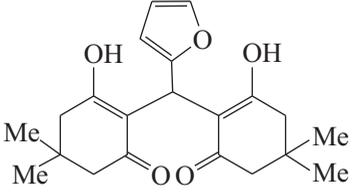
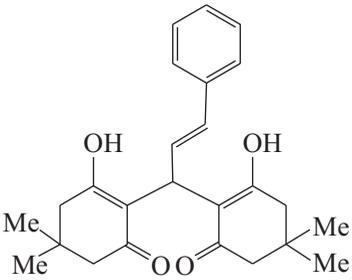
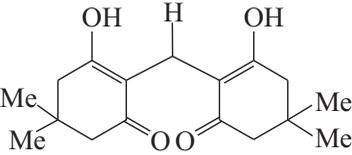
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**Table 2**  
Synthesis of tetraketone derivatives (Continued)

Entry	Products (3 a-r)	Yield (%)	Time (min)	Mp ( $^{\circ}$ C)	
				Found	Lit.
7	 <p><b>3g</b></p>	95	5	201–203	201–203 <sup>27</sup>
8	 <p><b>3h</b></p>	90	10	175–177	178–180 <sup>27</sup>
9	 <p><b>3i</b></p>	91	15	184–186	185–187 <sup>24</sup>
10	 <p><b>3j</b></p>	86	20	140–142	139–141 <sup>27</sup>

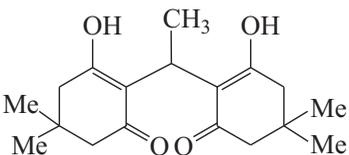
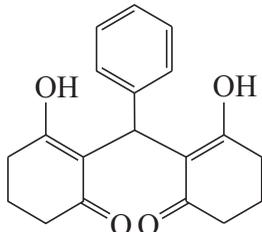
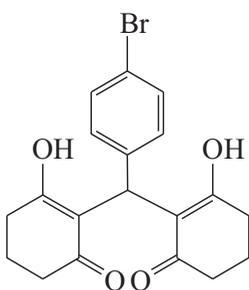
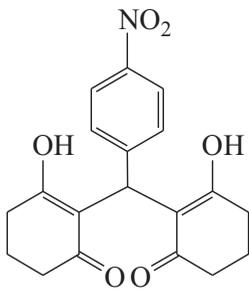
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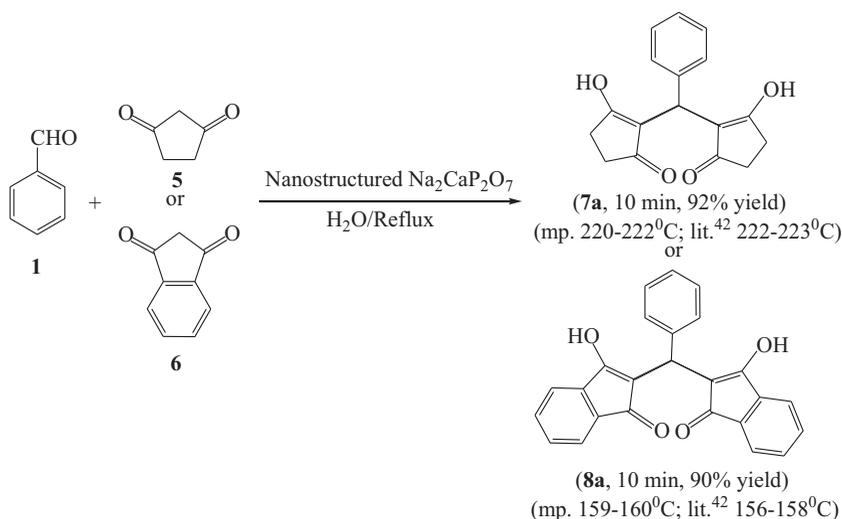
**Table 2**  
Synthesis of tetraketone derivatives (Continued)

Entry	Products (3 a-r)	Yield (%)	Time (min)	Mp ( $^{\circ}$ C)	
				Found	Lit.
11	 <p><b>3k</b></p>	87	20	193–195	195–196 <sup>28</sup>
12	 <p><b>3l</b></p>	90	10	140–142	139–141 <sup>27</sup>
13	 <p><b>3m</b></p>	86	25	203–205	204 <sup>27</sup>
14	 <p><b>3n</b></p>	84	40	191–193	192–193 <sup>30</sup>

(Continued on next page)

**Table 2**  
Synthesis of tetraketone derivatives (*Continued*)

Entry	Products (3 a-r)	Yield (%)	Time (min)	Mp ( $^{\circ}\text{C}$ )	
				Found	Lit.
15	 <p><b>3o</b></p>	81	50	182–183	182–184 <sup>30</sup>
16	 <p><b>3p</b></p>	92	10	206–208	207–208 <sup>30</sup>
17	 <p><b>3q</b></p>	91	10	238–240	240–241 <sup>31</sup>
18	 <p><b>3r</b></p>	92	5	193–195	194–196 <sup>28</sup>



**Scheme 2.** Reaction of benzaldehyde with 1,3-cyclopentadione or 1,3-indandione.

The reusability of the catalyst was also investigated. The same model reaction mentioned above was again studied under the optimized conditions. After the completion of the reaction, acetone was added to the reaction mixture to dissolve the product. Then the catalyst was removed by filtration and washed with acetone and calcined at 500 $^\circ\text{C}$  for 1 h before re-use. It was re-used directly in the model reaction to give **3 g** in yields of 95%, 92%, 89% for three consecutive runs at 5 min.

**Table 3**  
Comparison of the efficiency of various catalysts in the synthesis of tetraketone derivatives

Entry	Tetraketones	Conditions	Time (min)	Yield (%)
1	<b>3 a</b>	$\text{HClO}_4\text{-SiO}_2/\text{acetonitrile/reflux}$ [24]	360	54
		$\text{SmCl}_3/\text{water/rt}$ [27]	30	91
		$\text{KF}/\text{Al}_2\text{O}_3/\text{solvent-free/rt}$ [29]	840	86
		Catalyst-free/solvent-free/rt [30]	2880	86
		<i>p</i> -Dodecylbenzenesulfonic acid/water/us/25-30 $^\circ\text{C}$ [42]	60	89
		HFIP/ reflux [43]	180	90
		<b>Present work</b>	10	93
2	<b>3 g</b>	$\text{SmCl}_3/\text{water/rt}$ [27]	30	91
		PVP-Ni NPs/ ethylene glycol/rt [28]	10	90
		$\text{KF}/\text{Al}_2\text{O}_3/\text{solvent-free/rt}$ [29]	840	91
		Catalyst-free/water/rt [31]	2880	88
		$\text{Pd}(0)$ NPs/water/rt [38]	25	88
		<i>p</i> -Dodecylbenzenesulfonic acid/water/us/25-30 $^\circ\text{C}$ [42]	60	94
		HFIP/ reflux [43]	140	95
<b>Present work</b>	5	95		

In conclusion, novel synthetic nanostructured diphosphate ( $\text{Na}_2\text{CaP}_2\text{O}_7$ ) in the presence of water is an excellent catalyst for the synthesis of tetraketone derivatives *via* the condensation of aldehydes and 1,3-cyclic dicarbonyl compounds. The current method offers many advantages. These include high efficiency, high yields, short reaction times, freedom from waste, operational simplicity, facile separation and re-use of the catalyst from the reaction medium.

## Experimental Section

All reagents were obtained from commercial sources and were used without purification. IR spectra were recorded as KBr pellets on a Shimadzu 435-U-04 spectrophotometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were determined on a Bruker DRX-300 Avance spectrometer in  $\text{DMSO-d}_6$  or  $\text{CDCl}_3$ . Melting points were determined using an Electrothermal 9200 apparatus and are uncorrected. Scanning electron microscopy (SEM) images were obtained with a Philips  $\text{CM}_{10}$ . The synthesis of catalyst has been previously described.<sup>55-58</sup>

### *General Procedure for the Synthesis of Arylmethylene bis-(3-Hydroxy-2-cyclohexene-1-one)*

The catalyst,  $\text{Na}_2\text{CaP}_2\text{O}_7$  (20 mol%), was added to a mixture of the aldehyde (1 mmol), dimedone or 1,3-cyclohexandione (2 mmol), and 3 ml of water in a 5 ml flask fitted with a reflux condenser. The resulting mixture was heated to reflux for the appropriate time (*see Table 2*) with stirring. After the completion of the reaction as determined by TLC (silica gel; hexane-ethyl acetate, 4:1), acetone (2 ml) was added and the mixture stirred for 2 min. The catalyst was removed by filtration and washed with acetone and calcined at 500 °C for 1 h before re-use. After concentration of the filtrate, the residue was purified by recrystallization (8 ml, EtOH 96%) to afford the pure arylmethylene *bis*-(3-hydroxy-2-cyclohexene-1-one) derivatives. The products were identified by their melting points,  $^1\text{H}$ -NMR, and IR.

### *Spectral Data for Selected Compounds*

**2,2'-(Phenylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3 a):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.10 (s, 6 H, 2 $\text{CH}_3$ ), 1.23 (s, 6 H, 2 $\text{CH}_3$ ), 2.17–2.48 (m, 8 H, 4 $\text{CH}_2$ ), 5.54 (s, 1 H, CH), 7.08–7.28 (m, 5 H, Ar-H), 11.91 (s, 1 H, OH).

**2,2'-(4-Methoxyphenylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3b):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.02 (s, 6 H, 2 $\text{CH}_3$ ), 1.15 (s, 6 H, 2 $\text{CH}_3$ ), 2.21–2.40 (m, 8 H, 4 $\text{CH}_2$ ), 3.70 (s, 3 H,  $\text{OCH}_3$ ), 5.41 (s, 1 H, CH), 6.72–6.75 (d, 2 H, Ar-H), 6.93–6.91 (d, 2 H, Ar-H), 11.84 (s, 1 H, OH).

**2,2'-(4-Chlorophenylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3 d):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.02 (s, 6 H, 2 $\text{CH}_3$ ), 1.14 (s, 6 H, 2 $\text{CH}_3$ ), 2.21–2.41 (m, 8 H, 4 $\text{CH}_2$ ), 5.40 (s, 1 H, CH), 6.93–6.95 (d, 2 H, Ar-H), 7.14–7.17 (d, 2 H, Ar-H), 11.80 (s, 1 H, OH).

**2,2'-(4-Nitrophenylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3f):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.04 (s, 6 H, 2 $\text{CH}_3$ ), 1.16 (s, 6 H, 2 $\text{CH}_3$ ), 2.24–2.44 (m, 8 H, 4 $\text{CH}_2$ ), 5.57 (s, 1 H, CH), 7.03–7.51 (m, 4 H, Ar-H), 11.87 (s, 1 H, OH).

**2,2'-(3-Nitrophenylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3 g):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.05 (s, 6 H, 2 $\text{CH}_3$ ), 1.20 (s, 6 H, 2 $\text{CH}_3$ ), 2.24–2.45 (m, 8 H, 4 $\text{CH}_2$ ), 5.47 (s, 1 H, CH), 7.32–7.98 (m, 4 H, Ar-H), 11.79 (s, 1 H, OH).

**2,2'-(4-Methylphenylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3j):**  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.02 (s, 6 H, 2 $\text{CH}_3$ ), 1.15 (s, 6 H, 2 $\text{CH}_3$ ), 2.22 (s, 3 H,  $\text{CH}_3$ ), 2.25–2.40 (m, 8 H, 4 $\text{CH}_2$ ), 5.42 (s, 1 H, CH), 6.89–6.91 (d, 2 H, Ar-H), 6.99–7.19 (d, 2 H, Ar-H), 11.83 (s, 1 H, OH).

**2,2'-(Furan-2-ylmethylene)-bis-(3-hydroxy-5,5-dimethyl-2-cyclohexene-1-one) (3 l):**  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.10–1.18 (d, 12 H, 2 $\text{CH}_3$ ), 2.36 (s, 8 H, 4 $\text{CH}_2$ ), 5.39 (s, 1 H, CH), 5.95 (s, 1 H, CH), 6.30 (s, 1 H, CH), 7.26 (s, 1 H, CH), 12.17 (s, 1 H, OH).

**2,2'-(Phenylmethylene)-bis-(cyclopentane-1,3-dione) (7 a):**  $^1\text{HNMR}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  2.60–2.66 (m, 8 H, 4 $\text{CH}_2$ ), 5.37 (s, 1 H, CH), 7.11–7.24 (m, 5 H), 8.17 (brs, 2 H, OH).

**2,2'-(Phenylmethylene)-bis-(1 H-indene-1,3(2 H)-dione) (8 a):**  $^1\text{HNMR}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  3.94 (t, 1 H), 4.28 (d, 2 H), 7.19 (t, 1 H), 7.22 (t, 1 H), 7.38 (d, 2 H), 7.79–7.83 (m, 4 H), 7.89–7.94 (m, 4 H).

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