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## Study of in situ generation of carbocationic system from trityl chloride (Ph<sub>3</sub>CCl) which efficiently catalyzed cross-aldol condensation reaction

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## ABSTRACT

Organocatalyst trityl chloride (Ph<sub>3</sub>CCl), by in situ formation of trityl carbocation with inherent instability, efficiently promotes the cross-aldol condensation reaction between cycloalkanones and arylaldehydes in solvent-free and homogeneous media to afford α,α'-bis(arylidene)cycloalkanones in high yields. Moreover, an attractive and plausible mechanism based on observations and the literature is proposed for the reaction.

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### 1. Introduction

The use of small-molecule organocatalysts in organic synthesis has flourished over the past decade [1–9]. However, the concept of organocatalysis has emerged as a discrete strategy for addressing modern-day challenges in chemistry. It has become widely appreciated that small-molecule organic catalysts can hold a wide range of practical advantages relative to macromolecular, precious metal or protic acidic catalysts, including air stability, low cost, commercial availability, relative non-toxicity, green nature and simple reaction conditions, and can promote a chemical reaction through different activation modes [1–9]. With the dramatic recent expansion of research efforts in organocatalysis throughout the world, synthetically useful

transformations based on new reactivity concepts have been identified, often with no counterpart in the more established catalysis regimens [1–9]. Triarylmethyl chlorides (Ar<sub>3</sub>CCl) are an attractive class of small-molecule organocatalysts, which have been utilized to promote a few organic transformations by in situ formation of triaryl-methyl carbocations [6–9]. It is noteworthy that the inherent instability of carbocations has precluded up to now their use in catalysis with decent turnover numbers [7].

The aldol condensation reaction has been widely applied for carbon–carbon bond formation in organic synthesis [10–12]. Among them, the cross-aldol condensation of cycloalkanones with arylaldehydes leading to α,α'-bis(arylidene)cycloalkanones [13], which have attracted much attention due to their use as precursors for the synthesis of pyrimidine derivatives [14], and their intriguing biological activities such as antiangiogenic [15], quinine reductase inducer [16], and cholesterol-lowering properties [17]. Generally, the aldol condensation reaction

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has been catalyzed by strong acids [18] and more likely by bases [19]; however, in these conditions, the reactions suffer from reverse and/or side reactions [20]. Moreover, some metal ions with different ligands [21–25] and solid acids [26,27] have been introduced as catalysts to replace strong acids or bases. But most of these methods are associated with one or more of the following drawbacks: moderate yields, unwanted side reactions, need for application of sealed ampoules or tubes, use of expensive, unavailable and toxic catalysts, and application of an additional energy (microwave). Although some catalysts (mostly Brønsted and Lewis acids) for the cross-aldol condensation of cycloalkanones with arylaldehydes leading to  $\alpha,\alpha'$ -bis(arylidene)cycloalkanones are known, newer catalysts continue to attract attention for their difference with the others, high novelty and effectiveness.

In this work, we introduce a high novelty in the cross-aldol condensation reaction between cycloalkanones and arylaldehydes, and perform the reaction using trityl chloride ( $\text{Ph}_3\text{CCl}$ ) as an efficient and homogeneous small-molecule organocatalyst under solvent-free conditions at 110 °C. Attractive points of this work are study of in

**Table 1**

The reaction of cyclohexanone with *m*-nitrobenzaldehyde in the presence of different amounts of  $\text{Ph}_3\text{CCl}$  at various temperatures.

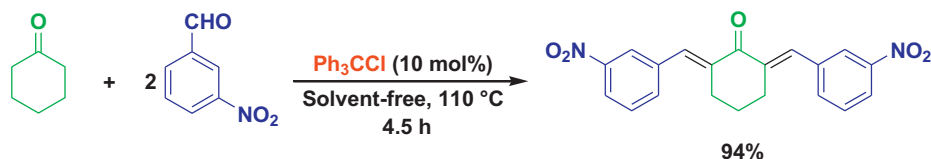
Entry	Mol% of $\text{Ph}_3\text{CCl}$	Temperature (°C)	Time (h)	Yield <sup>a</sup> (%)
1	–	110	8	19
2	7.5	110	6	76
3	10	110	4.5	94
4	12.5	110	4.5	92
5	10	100	4.5	83
6	10	120	4.5	90

<sup>a</sup> Isolated yield.

situ formation of trityl carbocation with inherent instability from trityl chloride ( $\text{Ph}_3\text{CCl}$ ) which catalyzes the reaction, and suggestion of a plausible mechanism based on observations and the literature.

## 2. Results and discussion

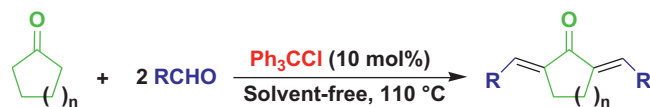
At first, the condensation of cyclohexanone (1 mmol) with *m*-nitrobenzaldehyde (2.1 mmol) was selected as a model reaction (Scheme 1), and its behavior was studied in



Scheme 1. The cross-aldol condensation between cyclohexanone and *m*-nitrobenzaldehyde catalyzed by  $\text{Ph}_3\text{CCl}$ .

**Table 2**

The solvent-free cross-aldol condensation of cycloalkanones with aldehydes using  $\text{Ph}_3\text{CCl}$  at 110 °C.



Entry	<i>n</i>	R	Time (h)	Yield <sup>a</sup> (%)	M.p. (°C)	
					Found	Reported
1	1	$\text{C}_6\text{H}_5$	4	87	186–188	187–189 [13]
2	1	<i>p</i> - $\text{MeOC}_6\text{H}_4$	4	92	207–209	210–211 [25]
3	1	<i>m</i> - $\text{ClC}_6\text{H}_4$	5.5	79	174–176	173–175 [13]
4	2	$\text{C}_6\text{H}_5$	5.5	87	113–115	116–117 [22]
5	2	<i>p</i> - $\text{NO}_2\text{C}_6\text{H}_4$	4	91	161–163	159 [21]
6	2	<i>m</i> - $\text{NO}_2\text{C}_6\text{H}_4$	4.5	94	184–186	180–182 [13]
7	2	<i>p</i> - $\text{MeOC}_6\text{H}_4$	4	88	165–167	160–162 [25]
8	2	<i>p</i> - $\text{MeC}_6\text{H}_4$	5.5	76	169–171	170 [22]
9	2	<i>p</i> - $\text{Me}_2\text{NC}_6\text{H}_4$	3.5	91	250–252	250–252 [30]
10	2	<i>p</i> - $\text{ClC}_6\text{H}_4$	4	90	143–145	147–148 [22]
11	2	<i>m</i> - $\text{ClC}_6\text{H}_4$	6	76	105–107	108–110 [13]
12	2	<i>o</i> - $\text{ClC}_6\text{H}_4$	5	84	104–106	103–105 [26]
13	2	2-Thienyl	5	92	141–143	142–143 [31]
14	2	2-Furyl	4.5	83	142–144	140–142 [13]
15	2	$\text{C}_6\text{H}_5\text{CH}=\text{CH}$	4.5	86	176–178	179–180 [25]
16	3	$\text{C}_6\text{H}_5$	6	86	104–106	105–107 [13]
17	3	3,4-( $\text{MeO}$ ) $_2\text{C}_6\text{H}_3$	3.5	89	165–167	162–164 [13]
18	3	<i>p</i> - $\text{ClC}_6\text{H}_4$	4	87	129–131	131–133 [13]

<sup>a</sup> Isolated yield.

the presence of different molar ratios of  $\text{Ph}_3\text{CCl}$  in the 100–120 °C range under solvent-free conditions. The results are displayed in Table 1. As it can be seen in Table 1, 10 mol% of the organocatalyst was sufficient to promote the reaction efficiently at 110 °C (Table 1, entry 3). Increasing the amount of  $\text{Ph}_3\text{CCl}$  or the temperature decreased the reaction yield (Table 1, entries 4 and 6). The reaction was also examined without catalyst in which the product was obtained in low yield even after a long reaction time (Table 1, entry 1).

After optimization of the reaction conditions, the efficiency, the generality and the scope of the organocatalyst in the preparation of  $\alpha,\alpha'$ -bis(arylidene)cycloalkanones were assessed by the reaction of cycloalkanones (cyclopentanone, cyclohexanone and cycloheptanone) with different arylaldehydes including benzaldehyde as well as aldehydes containing electron-withdrawing substituents, electron-releasing substituents or halogens (Table 2). As Table 2 indicates, all reactions proceeded efficiently and the desired products were obtained in high to excellent yields and in logical reaction times (Table 2, entries 1–12 and 16–18). Trityl chloride also successfully catalyzed the reaction of cyclohexanone with *p*-dimethylaminobenzaldehyde (containing a basic group), heteroaromatic aldehydes and cinnamaldehyde (Table 2, entries 9 and 13–15). Thus, in this reaction,  $\text{Ph}_3\text{CCl}$  was efficient and general catalyst.

Based on the literature reports related to the presented reaction shown in Scheme 2, our mechanistic proposal is that from the interaction of aldehyde and trityl chloride, tautomers **I** and **II** could be generated via a reversible reaction pathway [6–9].

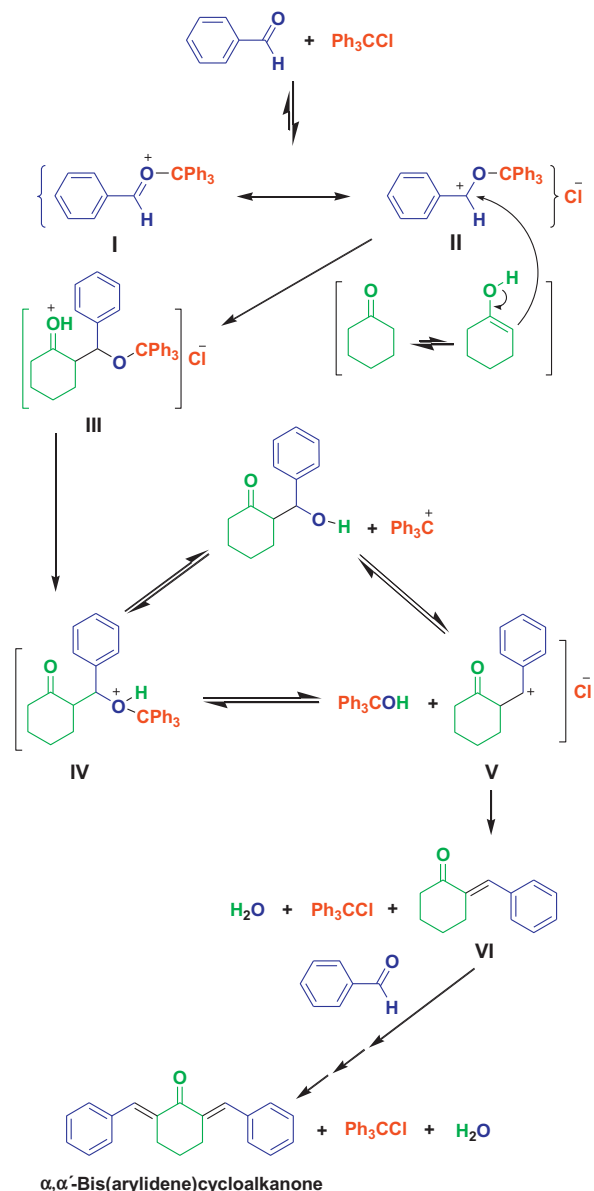
In order to validate our proposal, benzaldehyde was reacted with trityl chloride (in ratio of 1/1), and then IR,  $^1\text{H}$  NMR spectra of the resulting interacted aldehyde functional group of proposed tautomers **I** and **II** in the reaction mixture were compared with those in benzaldehyde as follows.

According to the obtained FT-IR spectrum of the resulting reaction mixture, stretching vibration of the carbonyl of the aldehyde functional group slightly shifts to lower frequency because of the increased dipole moment character of carbonyl functionality: IR (nujol):  $\nu_{\text{max}}$  ( $\text{cm}^{-1}$ ) of C=O in benzaldehyde (1705) decreased to (1697) in the reaction mixture (Fig. 1).

Accordingly, in the  $^1\text{H}$  NMR spectra, the aldehyde hydrogen as well as carbonyl carbon were deshielded, which is in agreement with the increased single bond character of carbonyl functional group observed in the FT-IR spectra:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) of the aldehydic hydrogen (9.78) increased to (10.04) in the reaction mixture (Fig. 2).

UV (*n*-hexane):  $\lambda_{\text{max}}$  (nm) of absorption of benzaldehyde and trityl chloride appears at 240 and 222, respectively; however,  $\lambda_{\text{max}}$  of the absorption of the complexes of benzaldehyde and trityl cation was observed at 245 (Fig. 3).

These results confirm that tautomer forms **I** and **II** are present in the reversible reaction media. Moreover, the cationic tautomers **I** and **II** have been introduced by Kusumoto et al. for the first time [28]. These tautomer forms act as activated aldehyde, and then react with



Scheme 2. The proposed mechanism for the cross-aldol condensation reaction using  $\text{Ph}_3\text{CCl}$ .

cyclohexanone providing **III**, which converts to **IV** by proton transfer. Intermediate **IV** can be converted to **V** by removing  $\text{Ph}_3\text{COH}$  in a reversible reaction. Afterward, **V** and  $\text{Ph}_3\text{COH}$  produce intermediate **VI**,  $\text{Ph}_3\text{CCl}$  and  $\text{H}_2\text{O}$ . **VI** accordingly reacts with another activated aldehyde by  $\text{Ph}_3\text{CCl}$  to afford the corresponding  $\alpha,\alpha'$ -bis(arylidene)cycloalkanone. The suggested mechanism is confirmed by the literature [29], and also by the fact that  $\text{Ph}_3\text{CCl}$  was completely recovered unchanged and  $\text{Ph}_3\text{COH}$  could not be identified after completion of the reaction as it could be observed on TLC by comparison with pure authentic samples. In another study, to demonstrate that  $\text{Ph}_3\text{CCl}$  cannot convert to  $\text{Ph}_3\text{COH}$  and  $\text{HCl}$  by the water produced during the reaction, and consequently  $\text{HCl}$  is not the real

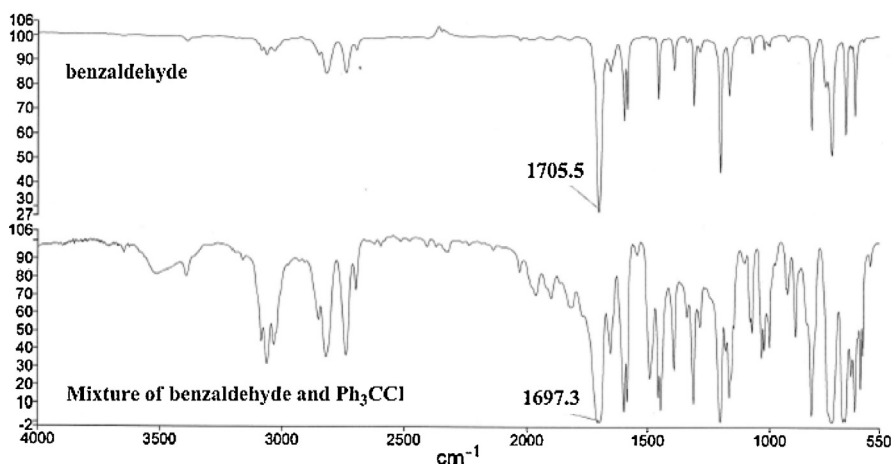


Fig. 1. IR spectra of benzaldehyde as well as the mixture of benzaldehyde and  $\text{Ph}_3\text{CCl}$  (in ratio of 1/1).

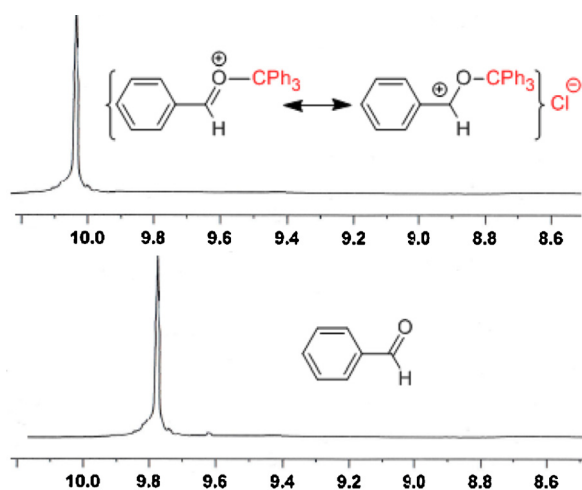


Fig. 2.  $^1\text{H}$  NMR spectra of the formyl hydrogen of benzaldehyde, separately as well as in the mixture of benzaldehyde and  $\text{Ph}_3\text{CCl}$  (in ratio of 1/1).

catalyst of the process, the solvent-free condensation of cyclohexanone with 3-nitrobenzaldehyde was examined in the presence of the expected amount of HCl produced by the reversible reaction of  $\text{Ph}_3\text{COH}$  with  $\text{H}_2\text{O}$ , at  $110^\circ\text{C}$ , in which the product was obtained in 48% within 8 h. The reaction was also tested using  $\text{Ph}_3\text{COH}$  wherein the reaction yield was 19% after 8 h. These evidences showed that HCl is not produced from  $\text{Ph}_3\text{CCl}$  in these conditions, and that  $\text{Ph}_3\text{CCl}$  is the real catalyst of this transformation.

### 3. Conclusion

In summary, we have shown that organocatalyst trityl chloride, by *in situ* generation of carbocationic system, efficiently catalyzed the cross-aldol condensation reaction. The promising points for the presented protocol are simple and clean reaction profile, efficiency, generality, high yields, reaction in solvent-free and homogeneous media without side reactions, economic availability,

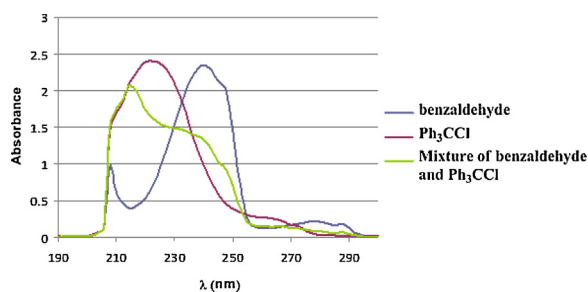


Fig. 3. UV spectra of benzaldehyde,  $\text{Ph}_3\text{CCl}$  and the complexes of benzaldehyde and  $\text{Ph}_3\text{CCl}$  (in ratio of 1/1).

non-toxicity as well as low cost of the catalyst, and good compliance with the green chemistry protocols which makes it an attractive procedure for the synthesis of  $\alpha,\alpha'$ -bis(arylidene)cycloalkanones as an important class of organic compounds.

## 4. Experimental

### 4.1. General

All chemicals were purchased from Merck or Fluka Chemical Companies. All known compounds were identified by comparison of their melting points and spectral data with those reported in the literature. Progress of the reactions was monitored by TLC using silica gel SIL G/UV 254 plates. The  $^1\text{H}$  NMR (500 MHz) and  $^{13}\text{C}$  NMR (125 MHz) were run on a Bruker Avance DPX, FT-NMR spectrometer. UV spectra were run on a T8 UV-VIS spectrometer, PG instruments Ltd. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

### 4.2. General procedure for the cross-aldol reaction

A mixture of cycloalkanone (1 mmol), aldehyde (2.1 mmol) and trityl chloride (0.028 g, 0.1 mmol) in a test tube connected to a reflux condenser, was stirred at  $110^\circ\text{C}$ ,

and the reaction progress was monitored by TLC. After completion of the reaction, the mixture was cooled to room temperature, petroleum ether (10 mL) was added to it, refluxed and stirred for 3 min, and filtered to separate the catalyst. The solid residue (crude product) was purified by recrystallization from EtOH (95%) or column chromatography on silica gel eluted with *n*-hexane/EtOAc (1/5) to give the pure  $\alpha,\alpha'$ -bis(arylidene)cycloalkanone.

#### 4.3. Selected spectral data of the products

**2,5-Bis(3-chlorobenzylidene)cyclopentanone** (Table 2, entry 3):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 3.01 (s, 4H), 7.29–7.33 (m, 4H), 7.39–7.42 (m, 2H), 7.43 (s, 2H), 7.51 (t,  $J$  = 1.2 Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 27.0, 128.6, 129.6, 130.5, 130.7, 132.5, 135.3, 137.1, 138.6, 196.0.

**2,6-Bis(3-chlorobenzylidene)cyclohexanone** (Table 2, entry 11):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.69 (quintet,  $J$  = 5.7 Hz, 2H), 2.78 (t,  $J$  = 5.7 Hz, 4H), 7.30–7.37 (m, 6H), 7.41 (s, 2H), 7.62 (t,  $J$  = 1.2 Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ): 23.7, 27.9, 126.3, 127.9, 129.3, 130.4, 130.9, 133.2, 135.8, 137.8, 188.9.

**2,6-Bis(2-chlorobenzylidene)cyclohexanone** (Table 2, entry 12):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.75 (quintet,  $J$  = 5.8 Hz, 2H), 2.78 (t,  $J$  = 5.8 Hz, 4H), 7.28 (m, 4H), 7.34 (d,  $J$  = 3.4, 2H), 7.45 (d,  $J$  = 3.3 Hz, 2H), 7.89 (t,  $J$  = 1.2 Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 23.2, 28.7, 126.8, 130.8, 130.9, 131.0, 134.5, 135.0, 135.5, 138.3, 189.2.

**(2E,6E)-2,6-Bis(E)-3-phenylallylidene)cyclohexanone** (Table 2, entry 15):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 1.85 (quintet,  $J$  = 5.8 Hz, 2H), 2.81 (t,  $J$  = 5.8 Hz, 4H), 6.97 (d,  $J$  = 15.4 Hz, 2H), 7.10 (dd, 2H,  $J$  = 15.4, 1.2 Hz), 7.28 (m, 2H), 7.36 (m, 4H), 7.48–7.54 (m, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 22.5, 27.8, 124.7, 124.8, 127.5, 129.3, 135.9, 136.8, 137.3, 141.4, 188.3.

**(2E,7E)-2,7-Dibenzylidenecycloheptanone** (Table 2, entry 16):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.03 (br, s, 4H), 2.75 (br, s, 4H), 7.37 (t,  $J$  = 7.2 Hz, 2H), 7.43 (s, 2H), 7.45 (m, 4H), 7.51 (d,  $J$  = 7.5 Hz, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 28.5, 29.3, 128.7, 128.9, 129.9, 136.1, 136.4, 142.2, 199.9.

**(2E,7E)-2,7-Bis(3,4-dimethoxybenzylidene)cycloheptanone** (Table 2, entry 17):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.05 (br, s, 4H), 2.77 (br, s, 4H), 3.91 (s, 6H), 3.96 (s, 6H), 6.94 (d,  $J$  = 8.5 Hz, 2H), 7.05 (s, 2H), 7.12 (d,  $J$  = 8.5 Hz, 2H), 7.39 (s, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ): 28.7, 29.1, 56.3, 56.4, 111.5, 113.3, 123.1, 129.2, 135.9, 140.5, 149.2, 149.7, 199.9.

**(2E,7E)-2,7-Bis(4-chlorobenzylidene)cycloheptanone** (Table 2, entry 18):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.00 (br, s, 4H), 2.71 (br, s, 4H), 7.38 (s, 2H), 7.41–7.45

(m, 8H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 28.4, 29.1, 129.2, 131.1, 134.6, 134.7, 135.0, 142.4, 199.3.

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