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FULL PAPER

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Sodium cyanide-promoted copper-catalysed aerobic oxidative synthesis of esters from aldehydes

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A simple and efficient copper-catalysed procedure for oxidative esterification of aldehydes with alcohols and phenols mediated by sodium cyanide, using air as a clean oxidant, is described. A variety of aromatic aldehydes and structurally different alcohols and phenols reacted efficiently, and the product esters were obtained in good to excellent yields under normal atmospheric and solvent-free conditions.

KEYWORDS

aerobic oxidation, alcohol, aldehyde, copper, ester, phenol

1 | INTRODUCTION

Esterification reaction is one of the most important reactions in organic synthesis that have been widely used in the chemical and pharmaceutical industries.[1] Conventionally, ester groups are synthesized by Fischer esterification which is a reversible reaction requiring acid catalyst[2] or by treatment of an alcohol with activated carboxylic acid derivatives in the presence of a stoichiometric amount of bases.[1] An alternative protocol involves transition-metal-catalysed coupling reactions. The first Pd-catalysed three-component esterification of aryl halides, alcohols and carbon monoxide was described by Heck and co-workers in 1974.[3] Since that pioneering report, several groups have studied these transformations using various Pd catalysts.[4] However, these procedures suffer from one or more disadvantages, such as the need for P ligands, elevated reaction temperatures and high pressure of toxic and harmful CO gas. Moreover, Pd is an expensive metal, and thus the Pd-based reactions are less attractive industrially. Some of these systems are also limited to using aryl iodides. In 2015, Iranpoor et al. reported the first $NiCl_2$ catalytic system for the alkoxycarbonylation of aryl iodides in the presence of $Cr(CO)_6$ as the solid source of carbon monoxide under air.[5] Though efficient, replacement of aryl halides as the coupling partners, which are generally environmental pollutants, is important. Therefore, there is still great interest in finding new methods and alternative coupling partners for the preparation of esters. Recently, the oxidative esterification of aldehydes has received growing attention and has become an economically attractive alternative to traditional ester synthesis. Direct oxidative transformation of an aldehyde moiety to an ester has been accomplished in a variety of ways.[6] Among these, coupling of aldehydes with alcohols in the presence of stoichiometric amounts of oxidants,[7] N-heterocyclic carbine activation[8] or transition-metal-mediated processes[9] are powerful strategies for this purpose. As an inexpensive, easily obtainable and environmental friendly catalyst, copper has been increasingly employed in many oxidative reactions.[10]

By taking these points into consideration and in continuation of our ongoing research interest in transition-metal-catalysed cross-coupling reactions,[11] we now introduce a one-pot procedure for Cu-catalysed preparation of esters from aldehydes and alcohols. To the best of our knowledge, a Cu-catalysed aerobic oxidative synthesis of esters through C─H activation of aldehydes has not been reported previously.

2 | RESULTS AND DISCUSSION

Initial studies were performed using the reaction of *p*-methylbenzaldehyde (1.0 mmol) with 2-phenylethanol (1.2 mmol) as the model reaction, employing CuI (20 mol%) as the catalyst and *tert*-butyl hydroperoxide (TBHP; 2.0 mmol) as oxidant in dimethylformamide (DMF) at 100°C under normal atmospheric conditions (Table 1, entry 1). In this case, the reaction did not proceed and no product was detected. It was further observed that increasing the catalyst loading up

TABLE 1 Effect of reaction parameters on Cu-catalysed synthesis of phenethyl 4-methylbenzoate[a,b]

Entry	Catalyst (mol%)	Oxidant (mmol)	Solvent/additive	Temp. (°C)	Yield (%)[c]
1	CuI (20)	TBHP (2.0)	DMF/—	100	—
2[d]	CuI (20)	TBHP (2.0)	DMF/NaCN	100	58
3	$Cu(OAc)_2$ (20)	TBHP (2.0)	DMF/NaCN	100	20
4	CuCl (20)	TBHP (2.0)	DMF/NaCN	100	40
5	$CuCl_2$ (20)	TBHP (2.0)	DMF/NaCN	100	20
6	$Cu(PPh_3)_3I$ (20)	TBHP (2.0)	DMF/NaCN	100	50
7	CuI (20)	TBHP (2.0)	DMSO/NaCN	100	Trace
8	CuI (20)	TBHP (2.0)	Toluene/NaCN	100	—
9	CuI (20)	TBHP (2.0)	MeCN/NaCN	Reflux	—
10	CuI (20)	TBHP (2.0)	THF/NaCN	Reflux	—
11[e]	CuI (20)	TBHP (2.0)	—/NaCN	100	67
12	CuI (20)	—	—/NaCN	100	65
13	CuI (20)	—	—/NaCN	120	91
14[f]	CuI (20)	—	—/NaCN	120	—
15	CuI (20)	—	—/NaCN (1.0 mml)	120	88
16	CuI (20)	—	—/NaCN (0.5 mmol)	120	88
17[g]	CuI (20)	—	—/NaCN (0.3 mmol)	120	45

[a] All reactions were carried out using 1.0 mmol of *p*-methylbenzaldehyde and 1.2 mmol of 2-phenylethanol.

[b] All reactions were worked up after 24 h.

[c] Isolated yield.

[d] 2.0 mmol of NaCN was added.

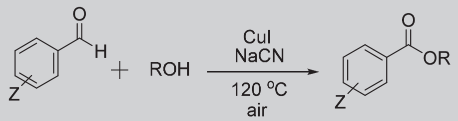
[e] Reaction was carried out under solvent-free conditions.

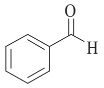
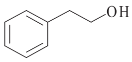
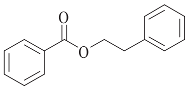
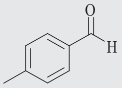
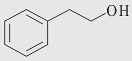
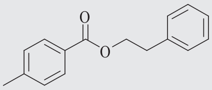
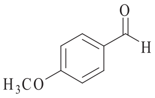
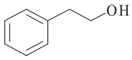
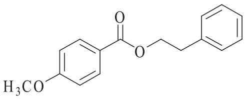
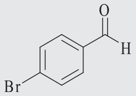
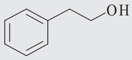
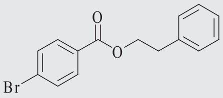
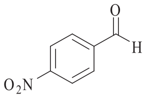
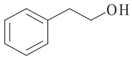
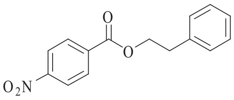
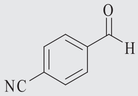
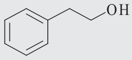
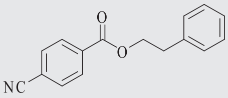
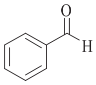
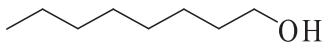
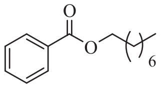
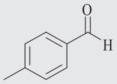
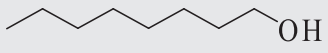
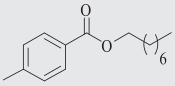
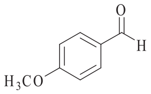
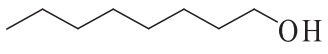
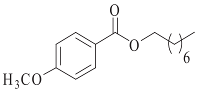
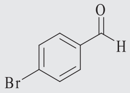
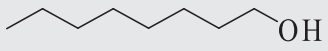
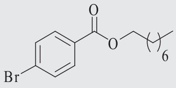
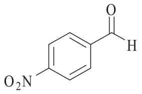
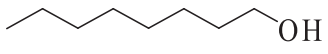
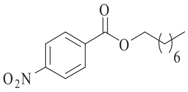
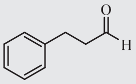
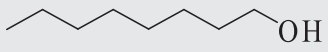
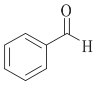
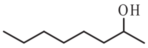
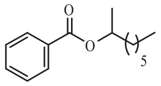
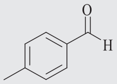
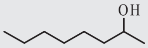
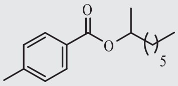
[f] Reaction was carried out under nitrogen atmosphere.

[g] Product was isolated after 48 h.

to 50 mol% did not have any effect on the progress of the reaction. Similarly, increasing the amount of TBHP failed to give any improvement. Inspired by the oxidation of cyanohydrins with manganese dioxide of Corey et al.,[7] a similar reaction in the presence of NaCN was studied. Surprisingly, we found that by addition of 2.0 mmol of sodium cyanide to the reaction mixture, the reaction proceeded and afforded the desired product after 24 h, albeit in moderate yield (Table 1, entry 2). This promising result encouraged us to improve the yield of the product. Performing the reaction in the presence of other Cu-based catalysts such as $Cu(OAc)_2$, CuCl, $CuCl_2$ and $Cu(PPh_3)_3I$ showed that the appropriate choice of catalyst was CuI (Table 1, entries 3–6). Screening of solvents under identical conditions revealed that DMF was the most effective solvent in this coupling reaction, whereas other solvents such as dimethylsulfoxide (DMSO), toluene, MeCN and tetrahydrofuran (THF) were less effective (Table 1, entries 7–10). Next, we carried out the reaction under solvent-free conditions at 100°C. Interestingly, this process provided the coupling product in better yield (Table 1, entry 11). Disposal of organic solvents is a major problem for the chemical industry. Furthermore, organic solvents are expensive, toxic and flammable and are not recyclable for the most part. Therefore, the subsequent reactions were performed under solvent-free conditions. Moreover, it was interesting to find that when the TBHP as oxidant was omitted from the reaction mixture, essentially identical results were obtained (Table 1, entry 12). This result suggested that atmospheric oxygen played a significant role in the reaction. Decrease in the amount of the catalyst showed a longer reaction time along with a lower yield and increase in the catalyst loading beyond 20 mol% did not increase the product yield appreciably. Control experiments indicated that no reaction took place in the absence of the catalyst. The reaction temperature played an important role in this transformation. A clear improvement of the yields was observed when the reaction temperature was raised from 100 to 120°C (Table 1, entry 13). Further increase in the reaction temperature did not affect the reaction appreciably. As expected, when the reaction was carried out under nitrogen atmosphere, no product was detected (Table 1, entry 14). On the other hand, the reaction yield of the model reaction was decreased

TABLE 2 Cu-catalysed oxidative esterification of aldehydes with various alcohols[a]



Entry	Aldehyde	Alcohol	Product	Time (h)	Yield (%)[b]
1				23	91[12]
2				24	88[13]
3				48	75[13]
4				25	90[13]
5				18	92[13]
6				20	88[13]
7				28	82[14]
8				33	86[14]
9				48	77[5]
10				25	93[14]
11				17	89[15]
12			—	48	—
13				32	85[5]
14				35	80[16]

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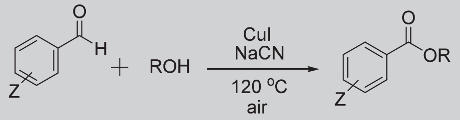
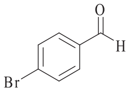
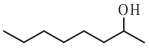
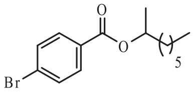
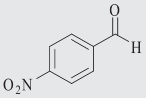
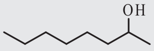
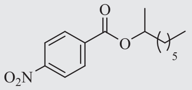
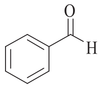
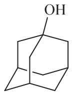
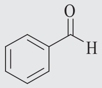
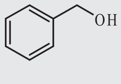


Entry	Aldehyde	Alcohol	Product	Time (h)	Yield (%)[b]
15				32	83[16]
16				20	91[16]
17			—	24	—
18[c]			—	24	—

[a]Reaction conditions: aldehyde (1.0 mmol), alcohol (1.2 mmol), NaCN (0.5 mmol), CuI (20 mol%) at 120°C.

[b]Isolated yield.

[c]Benzaldehyde was obtained under the optimum conditions.

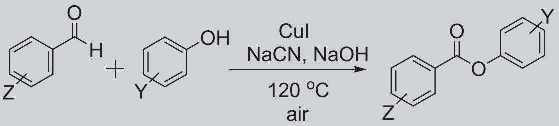
from 91 to 75% by blowing air into the reaction mixture at a rate of about 5 ml min^{-1} using an air pump probably due to the escape of organic reactants and product molecules at 120°C. After that, the required amount of cyanide ion was optimized. The results of Table 1 show that decreasing the amount of NaCN from 2.0 to 0.5 eq. caused no effect on yield and reaction time, but lower amounts of cyanide ion not only extended the reaction time, but also greatly reduced the reaction yield (Table 1, entries 15–17). Therefore, the optimized reaction conditions are as follows: reacting 1.0 mmol of *p*-methylbenzaldehyde with 1.2 mmol of 2-phenylethanol and 0.5 mmol of NaCN in the presence of 20 mol% of CuI at 120°C.

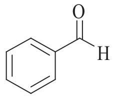
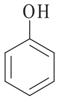
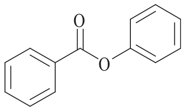
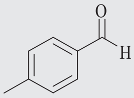
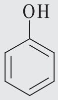
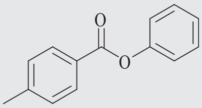
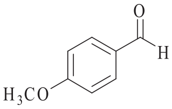
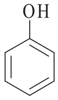
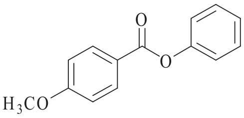
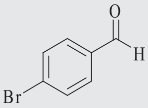
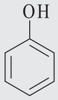
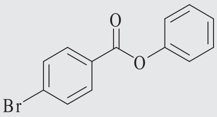
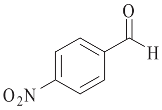
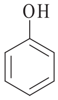
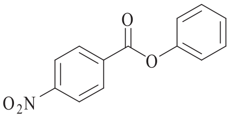
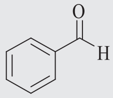
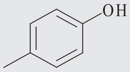
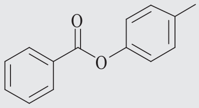
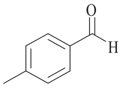
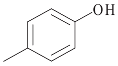
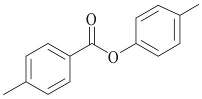
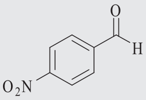
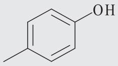
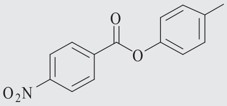
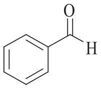
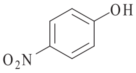
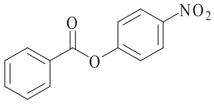
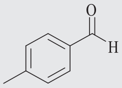
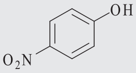
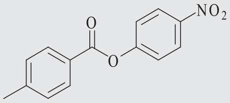
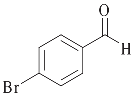
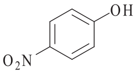
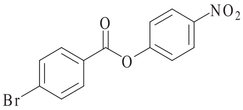
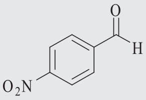
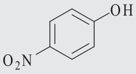
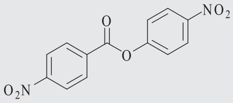
With the optimized conditions in hand, a broad range of substituted aldehydes were subjected to esterification with 2-phenylethanol and 1-octanol. As evident from Table 2, all reactions with aromatic aldehydes proceeded smoothly in good to excellent yields, and various functional groups, such as methyl, methoxy, nitro and bromo groups, on the phenyl ring of aldehydes were tolerated, whereas aliphatic aldehydes did not react under the reaction conditions (Table 2, entry 12). Next, this protocol was extended to different alcohols. As evident from Table 2, this reaction is not limited to primary alcohols. 2-Octanol as a secondary alcohol also afforded the desired products in good to excellent yields when it was treated with structurally diverse aldehydes under the reaction conditions. The reaction with primary alcohols was slightly faster in comparison with the reactions when bulkier secondary ones were used (Table 2, entries 7 and 13). Tertiary alcohol did not undergo esterification (Table 2, entry 17), probably due to steric effects. It is also noteworthy that the esterification of benzylic alcohols did not occur under the reaction conditions. In this case, oxidation worked as a competing reaction giving benzaldehydes.

Despite extensive efforts in the synthesis of simple esters, less attention has been paid to the synthesis of aryl benzoate derivatives. Aryl benzoates are important compounds with wide-ranging applications in organic and bioorganic chemistry.[17,18] The traditional syntheses of these compounds involve the reaction of phenols with activated carboxylic acid derivatives,[19] transesterification[20] and Baeyer–Villiger oxidation reactions.[21] These reactions often require strong acidic or basic conditions, which limit the substrate scope. Recently, efforts have been devoted towards the oxidative coupling between aldehydes and phenols.[22] However, in many cases, a carbonyl group at the *ortho* position of the phenolic function is essential for successful performance of the reaction.[23]

Encouraged by the results obtained in the transformation of aldehydes to esters with alcohols, we decided to investigate the applicability of performing this protocol using phenols as coupling partners. Therefore, we ran the reaction of *p*-methylbenzaldehyde with phenol under the optimal conditions of ester formation with alcohols. In this case, the reaction gave phenyl 4-methylbenzoate in only 30% yield after 48 h. To improve the reaction yield we screened other solvents, oxidants and amount of phenol or copper salt in the model reaction, but they were found to be less effective.

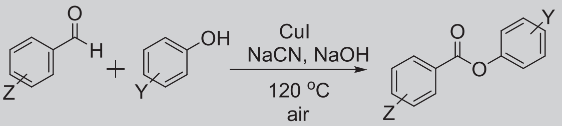
TABLE 3 Cu-catalysed synthesis of benzoate esters via oxidative coupling of aldehydes with phenols[a]



Entry	Aldehyde	Phenol	Product	Time (h)	Yield (%)[b]
1				23	91[5]
2				26	88[18]
3				31	84[18]
4				20	91[18]
5				15	92[18]
6				23	85[15]
7				25	92[24]
8				15	90[15]
9				48	75[15]
10				48	79[22]
11				48	81[22]
12				31	93[25]

(*Continues*)

TABLE 3 (Continued)



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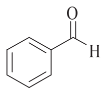
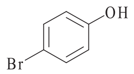
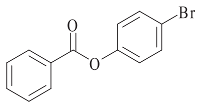
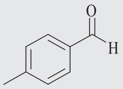
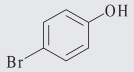
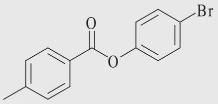
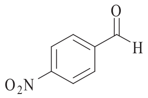
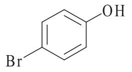
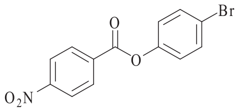
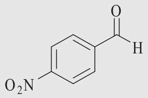
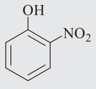
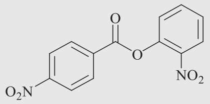
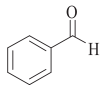
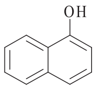
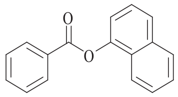
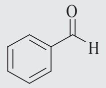
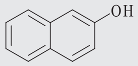
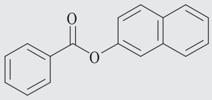
13				25	84[22]
14				28	88[26]
15				18	90[26]
16				48	73[27]
17				48	79[5]
18				23	89[18]

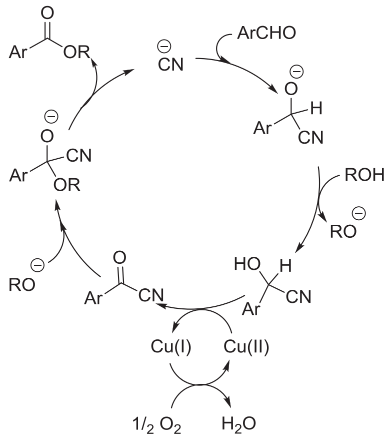
[a] Reaction conditions: aldehyde (1.0 mmol), phenol (1.2 mmol), NaCN (0.5 mmol), CuI (20 mol%), NaOH (1.2 mmol) at 120°C.

[b] Isolated yield.

To our delight, a clear improvement of the yield was observed when 1.2 mmol of NaOH as base was added to the reaction mixture. Having these results in hand, we were able to extend the applicability of this protocol for the oxidative coupling of substituted benzaldehydes with a range of phenols (Table 3). Various benzaldehydes, including those containing electron-withdrawing and electron-releasing groups, were readily converted to the corresponding products in excellent yields. Substituted phenols were also tested in the reaction with aldehydes and the desired aryl esters were efficiently formed.

Generally, the reaction of electron-rich phenols with aldehydes was found to be completed in shorter time than that of electron-deficient phenols (e.g. Table 3, entries 6 and 8 versus 9 and 12). On the other hand, the electron-rich aldehydes provided the desired products slower than electron-poor ones (Table 3, entries 1–5). The steric hindrance of phenols had a sharp effect on the reaction. For example, 93% esterified product was isolated within 31 h when 4-nitrophenol was used as the substrate in reaction with 4-nitrobenzaldehyde (Table 3, entry 12), while 2-nitrophenyl-4-nitrobenzoate was obtained in 73% yield after 48 h, in the case of 2-nitrophenol (Table 3, entry 16).

Based on our experimental data and the literature, we propose the following catalytic cycle for aerobic oxidation of aldehydes with alcohols (Scheme 1). Since the catalytic activity is not evident in the absence of cyanide ion, in the first step, formation of cyanohydrin from the reaction of aldehydes with sodium cyanide is expected. Subsequently, the formed



SCHEME 1 Proposed mechanism for aerobic oxidative synthesis of esters

cyanohydrin is converted to an acyl cyanide intermediate in the presence of Cu(II) species (generated *in situ* from aerobic oxidation of Cu(I)). Finally, the nucleophilic addition of alcohols on acyl cyanide intermediate affords the desired ester and Cu(I) catalyst which can be re-oxidized to Cu(II) by air.

3 | CONCLUSIONS

We described a cu-catalysed aerobic oxidative esterification of aldehydes with alcohols and phenols under solvent-free conditions mediated by sodium cyanide. oxygen as a clean oxidant was used in this simple one-pot process, producing esters in good to excellent yields. although nacn is toxic, the presented procedure obviates the employment of stoichiometric amounts of oxidants or environmental contaminant aryl halides. the system was selective for primary and secondary alcohols and was not suitable for the use of tertiary alcohols and benzylic alcohols as nucleophiles.

4 | EXPERIMENTAL

4.1 | General Procedure for Cu-Catalysed Esterification of Aldehydes with Alcohols

To a 25 ml round-bottom flask equipped with a magnetic stirrer bar and a condenser, an aldehyde (1.0 mmol), an alcohol (1.2 mmol), NaCN (0.5 mmol, 0.024 g) and CuI (20 mol%, 0.038 g) were added. The reaction mixture was heated in an oil bath at 120°C. The progress of the reaction was monitored by TLC. After the completion of the reaction, the mixture was cooled to room temperature and the product was extracted with ethyl acetate (3 × 3 ml). The solvent was then evaporated to leave the crude product, which was purified by column chromatography over silica gel using *n*-hexane as eluent to afford the highly pure product. The products were identified by their spectral data and comparison with authentic samples.

4.2 | General Procedure for Cu-Catalysed Esterification of Aldehydes with Phenols

CuI (20 mol%, 0.038 g) was added to a 25 ml round-bottom flask containing an aldehyde (1.0 mmol), a phenol (1.2 mmol), NaCN (0.5 mmol, 0.024 g) and NaOH (1.2 mmol, 0.048 g). The flask was then equipped with a condenser and the mixture was stirred at 120°C in an oil bath using a magnetic stir bar. After completion of the reaction, which was monitored by TLC, the mixture was cooled to room temperature and the esterified product was extracted with ethyl acetate (3 × 3 ml). The solvent was then evaporated to leave the crude product, which was purified by column chromatography over silica gel using *n*-hexane–ethyl acetate (10:1) as the eluent to give pure product. The products were identified by their spectral data and comparison with authentic samples.

4.3 | Typical Reaction of *p*-Methylbenzaldehyde, 2-Phenylethanol and NaCN in Presence of CuI under Nitrogen Atmosphere

A 50 ml two-neck flask containing *p*-methylbenzaldehyde (1.0 mmol), 2-phenylethanol (1.2 mmol), NaCN (0.5 mmol, 0.024 g) and CuI (20 mol%, 0.038 g) was fitted with a gas inlet and a magnetic stir bar. A condenser equipped with a balloon was attached, and the apparatus was evacuated before filling with nitrogen. The mixture was stirred at 120°C for 24 h. After that, the mixture was cooled to room temperature and extracted with ethyl acetate (3 × 3 ml). The extract was analysed by TLC. No product was detected. However, the organic mixture was subjected to column chromatography over silica gel using 10:1 *n*-hexane–ethyl acetate as eluent to separate unreacted *p*-methylbenzaldehyde and 2-phenylethanol, separated in, respectively, 85 and 94% yields.

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