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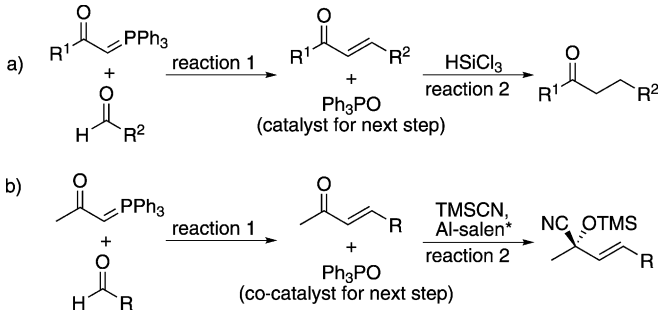
Tandem One-Pot Wittig/Reductive Aldol Reactions in which the Waste from One Process Catalyzes a Subsequent Reaction

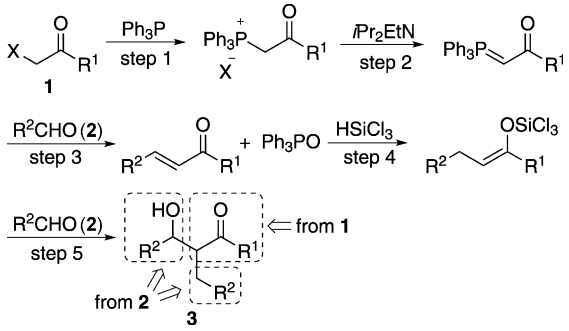
Jinni Lu and Patrick H. Toy*[a]

Tandem reactions, in which the product of one reaction immediately serves as the starting material for a subsequent transformation, have become attractive in organic synthesis as they allow for the efficient build-up in structural complexity from relatively simple starting materials in multistep, one-pot procedures.[1] Usually, such processes are designed so that only a single reagent or catalyst is required to perform all of the desired steps of the reaction sequence, and thus issues regarding compatibility are sidestepped. However, Zhou and co-workers have very recently described a conceptual breakthrough in this context in which the byproduct of one reaction serves as the catalyst for the second reaction in one-pot reaction sequences.[2] Specifically they reported tandem Wittig–conjugate-reduction reaction cascades in which the Ph_3PO generated as waste in the first process catalytically activates $HSiCl_3$ in the second step (Scheme 1a). Furthermore, they also found that the Ph_3PO formed in Wittig reactions could work together with a chiral salen aluminium complex in catalytic asymmetric cyanosilylation reactions of enones formed in the first reaction (Scheme 1b).



Scheme 1. The "waste as catalyst/co-catalyst" strategy.

While the Wittig reaction has been a workhorse for alkene synthesis since its discovery over half a century ago,[3,4] its deficiencies with regard to atom economy[5] and product purification are well known.[6] In relation to the latter point, we have been studying polymer-supported phosphines,[7–9] and have recently reported new phosphine-functionalized polystyrenes that can be used in one-pot Wittig reactions in which product isolation/purification is reduced to simple filtration and solvent removal operations.[10,11] The one-pot reactions studied involved in situ generation of the required phosphorane by mixing the polymer-supported phosphine reagent, an α-halo carbonyl compound, and an amine base, followed by a reaction with an aldehyde substrate.[12] As the methodology reported by Zhou and co-workers used preformed phosphoranes, we were inspired to see if it would be possible to couple one-pot Wittig reactions with the phosphine oxide-catalyzed[13,14] reductive aldol reactions reported by Suigura and Nakajima[15–18] in reaction sequences involving five distinct steps: 1) phosphorous alkylation, 2) phosphonium salt deprotonation, 3) Wittig reaction, 4) conjugate hydride addition, and 5) an aldol reaction (Scheme 2). From the perspective of diversity-oriented synthesis, such methodology would be highly attractive as it



Scheme 2. The five-step one-pot Wittig/reductive aldol reaction cascade concept.

[a] J. Lu, Prof. Dr. P. H. Toy
Department of Chemistry
The University of Hong Kong
Pokfulam Road, Hong Kong (P. R. of China)
Fax: (+852) 2857-1586
E-mail: phtoy@hku.hk

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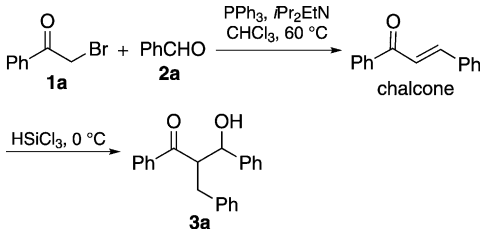
could allow for a large number of activated alkyl halides (**1**) and nearly all aldehydes (**2**), or a combination of aldehydes (when **2** is different in steps 3 and 5), to be combined in a simple, one-pot procedure to generate products of the general structure **3**. Herein we report the realization of this concept.

In the original report regarding asymmetric phosphine oxide-catalyzed reductive aldol reactions, it was observed that chalcone was a good substrate in such processes.[15b] Thus, as we previously found that 2-bromoacetophenone (**1a**) was a suitable activated alkyl halide for one-pot Wittig reactions with benzaldehyde (**2a**),[10a] we chose this substrate as a starting point to study the reaction involving these two building blocks (Scheme 3). As one equivalent of **2a** would

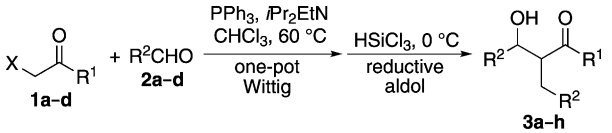


Scheme 3. Tandem chalcone synthesis/reductive aldol reaction.

react with the phosphorane to form chalcone prior to the reductive aldol reaction, we used a 1:1:1:2.2 molar ratio of **1a**/Ph_3P/*i*Pr_2EtN/**2a** in our preliminary experiments. Gratifyingly, in chloroform at 60 °C, the initial Wittig reaction to form chalcone was completed within 2 hours. The reaction mixture was cooled to 0 °C and then $HSiCl_3$ (2 equivalents) was added. After one hour at this temperature, the in situ formed chalcone was completely consumed, and **3a** was isolated in 67 % yield as a 34:66 mixture of *syn*/*anti* diastereomers (Table 1, entry 1). While at first glance the yield may not appear to be overly impressive, it should be noted that 67 % represents the overall yield of five sequential transformations (i.e. approximately 92 % yield per step), and as large excesses of reagents were not used, purification of the desired product **3a** was relatively straightforward. Furthermore, the observed stereoselectivity of the reductive aldol step was similar to what was previously observed when Ph_3PO was used as the catalyst in similar reactions.[15a]

Encouraged by this positive result, we next examined the substrate scope of the tandem one-pot Wittig/reductive aldol condensation reaction procedure. Other aryl aldehydes, such as 4-bromobenzaldehyde (**2b**) and 4-methylbenzaldehyde (**2c**), were found to work well with **1a**, and afforded a high overall yield of **3b** and **3c**, respectively (Table 1, entries 2 and 3). However, when branched isobutyraldehyde (**2d**) was used with **1a**, only a low yield of the desired product **3d** was obtained, even when the reductive aldol reaction was performed at room temperature (Table 1, entry 4). When acetophenones bearing aryl substituents, such as 2-bromo-4′-bromoacetophenone (**1b**) and 2-bromo-4′-methoxyacetophenone (**1c**), were used as the alkyl halide with **2a**, **3e** and **3f** were obtained in excellent yield, respectively (Table 1, entries 5 and 6). Additionally, chloroacetone (**1d**) was also found to be a suitable alkyl halide, and when used with **2a** or **2b**, products **3g** or **3h** were isolated in 48 % or 62 % overall yield, respectively (Table 1, entries 7 and 8). In these reactions, the general trend that the *anti* isomer of the product was slightly favored was observed, as 1:1–1:3 ratios of *syn*/*anti* stereoisomers of **3a**–**h** were obtained. As already mentioned, such stereoselectivity mirrors what has been reported in the literature.[15a]

Table 1. Tandem one-pot Wittig/reductive aldol reactions using a single aldehyde.



1a: X = Br, R^1 = Ph
1b: X = Br, R^1 = 4-BrC_6H_4
1c: X = Br, R^1 = 4-$MeOC_6H_4$
1d: X = Cl, R^1 = Me

2a: R^2 = Ph
2b: R^2 = 4-BrC_6H_4
2c: R^2 = 4-MeC_6H_4
2d: R^2 = *i*Pr

Entry	1	2	3	Yield [%][a]	*syn*/*anti*
1	**1a**	**2a**	**3a** R^1 = Ph, R^2 = Ph	67	34:66
2	**1a**	**2b**	**3b** R^1 = Ph, R^2 = 4-BrC_6H_4	81	41:59
3	**1a**	**2c**	**3c** R^1 = Ph, R^2 = 4-MeC_6H_4	73	34:66
4	**1a**	**2d**	**3d** R^1 = Ph, R^2 = *i*Pr	12[b]	39:61
5	**1b**	**2a**	**3e** R^1 = 4-BrC_6H_4, R^2 = Ph	82	41:59
6	**1c**	**2a**	**3f** R^1 = 4-$MeOC_6H_4$, R^2 = Ph	85	24:76
7	**1d**	**2a**	**3g** R^1 = Me, R^2 = Ph	48	33:67
8	**1d**	**2b**	**3h** R^1 = Me, R^2 = 4-BrC_6H_4	62	32:68

[a] Yield of the isolated product of reactions using **1a**–**d** (0.5 mmol), **2a**–**d** (1.1 mmol), PPh_3 (0.5 mmol), *i*Pr_2EtN (0.5 mmol), and 4 Å molecular sieves (0.1 g) in $CHCl_3$ (1 mL) stirred at 60 °C for 2–24 h, followed by the addition of $HSiCl_3$ (1.0 mmol) and stirring at 0 °C for 1–4 h. The reaction products were isolated as diastereomeric mixtures if not separable. [b] The reductive aldol reaction was performed at room temperature.

As a strategy to increase the structural diversity of the products **3**, we next considered the possibility of using one aldehyde in the one-pot Wittig reaction and a second, different aldehyde as the electrophile in the reductive aldol process. Thus, for the next set of experiments the aldehyde used in the one-pot Wittig reaction, **2a** or 3-phenylpropanal (**2e**), was used as the limiting reagent, rather than in excess as before (Table 2). For example, a mixture of **1a**, **2a**, Ph_3P, and *i*Pr_2EtN was heated to 60 °C until **2a** was completely consumed, and then the reaction was cooled to 0 °C and $HSiCl_3$ (2 equivalents) and 4-nitrobenzaldehyde (**2f**; 1.2 equivalents) were added to eventually form **3i** in 57 % yield (Table 2, entry 1). Other arylaldehydes, such as benzaldehydes **2g**–**i** and heteroaromatic **2j**, could also be used in the reductive aldol step with in situ generated chalcone to afford **3j**–**m** in high overall yields (Table 2, entries 2–5). When the initial one-pot Wittig reaction was performed using **1a** and aliphatic aldehyde **2e**, and **2a** was used in the reductive aldol reaction, **3m** was isolated in 71 % overall yield (Table 2, entry 6). Finally, using **1d** in the first step also afforded good results with various aldehyde combinations (Table 2, entries 7 and 8). The stereoselectivity ob-

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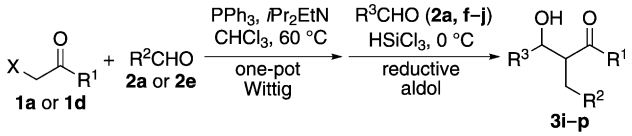
Table 2. Tandem one-pot Wittig/reductive aldol reactions using two aldehydes.



1a: X = Br, R^1 = Ph
1d: X = Cl, R^1 = Me

2a: R^2 = Ph
2e: R^2 = $PhCH_2CH_2$
2f: R^2 = 4-$NO_2C_6H_4$
2g: R^2 = 4-FC_6H_4
2h: R^2 = 4-ClC_6H_4
2i: R^2 = 4-$MeOC_6H_4$
2j: R^2 = 2-furyl

Entry	1	1st aldehyde	2nd aldehyde	3		Yield [%][a]	*syn*/*anti*
1	**1a**	**2a**	**2f**	**3i**	$R^1=R^2=$Ph, $R^3=$4-$NO_2C_6H_4$	57	51:49
2	**1a**	**2a**	**2g**	**3j**	$R^1=R^2=$Ph, $R^3=$4-FC_6H_4	68	45:55
3	**1a**	**2a**	**2h**	**3k**	$R^1=R^2=$Ph, $R^3=$4-ClC_6H_4	78	37:63
4	**1a**	**2a**	**2i**	**3l**	$R^1=R^2=$Ph, $R^3=$4-$MeOC_6H_4$	68	29:71
5	**1a**	**2a**	**2j**	**3m**	$R^1=R^2=$Ph, $R^3=$2-furyl	67	50:50
6	**1a**	**2e**	**2a**	**3n**	$R^1=$Ph, $R^2=PhCH_2CH_2$, $R^3=$Ph	71	25:75
7	**1d**	**2a**	**2h**	**3o**	$R^1=$Me, $R^2=$Ph, $R^3=$4-ClC_6H_4	49	34:66
8	**1d**	**2e**	**2a**	**3p**	$R^1=$Me, $R^2=PhCH_2CH_2$, $R^3=$Ph	60	31:69

[a] Yield of the isolated product of reactions using **1a** or **1d** (0.65 mmol), **2a** or **2e** (0.5 mmol), PPh_3 (0.65 mmol), *i*Pr_2EtN (0.65 mmol), and 4 Å molecular sieves (0.1 g) in $CHCl_3$ (1 mL) stirring at 60 °C for 16–24 h, followed by the addition of **2a**, **2f–j** (0.6 mmol) and $HSiCl_3$ (2.0 mmol) stirring at 0 °C for 1–2.5 h. The reaction products were isolated as diastereomeric mixtures if not separable.

served in this set of reactions involving two aldehydes was similar to what was found before when one aldehyde was used, and products **3i–p** were formed as approximately 1:1–1:3 mixtures of *syn*/*anti* stereoisomers.

In summary, we have developed a tandem one-pot Wittig/reductive aldol reaction procedure that allows for the rapid synthesis of complex molecules that are composed of three building blocks. This protocol is noteworthy in that the by-product of the one-pot Wittig reaction, Ph_3PO, catalyzes the subsequent reductive aldol process. Furthermore, the flexibility of this strategy is exhibited by the fact that different aldehydes can be used in the one-pot Wittig and reductive aldol reactions. Thus, it appears to have potential in the context of diversity-oriented synthesis.

As Ph_3PO is a byproduct of several reactions, including the Mitsunobu reaction,[19] and is a good Lewis base catalyst for numerous processes,[14] this general strategy for performing tandem reaction sequences could have many applications in organic synthesis. We are currently investigating such synthetic procedures, which include chiral phosphines and enantioselective phosphine oxide catalysis, and will report our findings in due course.

Experimental Section

General Procedure A for Tandem One-Pot Wittig/Reductive Aldol Reactions using a Single Aldehyde (Table 1)

A mixture of alkyl halide **1a–d** (0.5 mmol), aldehyde **2a–d** (1.1 mmol), PPh_3 (0.5 mmol), *i*Pr_2EtN (0.5 mmol), and 4 Å molecular sieves (0.1 g) in $CHCl_3$ (1 mL) was stirred at 60 °C for 2–24 h. Then $HSiCl_3$ (1.0 mmol) was added at 0 °C and the reaction mixture was stirred for 1–4 h more. The reaction was quenched with sat. $NaHCO_3$, filtered through Celite, and washed with ethyl acetate. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. The crude product was purified by silica-gel column chromatography (ethyl acetate/hexane = 1:15 then 1:3) to afford *syn*- and *anti*-**3a–h** as pure stereoisomers, or as a mixture of diastereomers.

General Procedure B for Tandem One-Pot Wittig/Reductive Aldol Reactions using Two Different Aldehydes (Table 2)

A mixture of alkyl halide **1a** or **1d** (0.65 mmol), aldehyde **2a** or **2e** (0.5 mmol), PPh_3 (0.65 mmol), *i*Pr_2EtN (0.65 mmol), and 4 Å molecular sieves (0.1 g) in $CHCl_3$ (1 mL) was stirred at 60 °C for 16–24 h until complete consumption of the aldehyde was observed by TLC analysis. Aldehydes **2f–j** (0.6 mmol) and $HSiCl_3$ (1.0 mmol) were then added at 0 °C. After stirring for 1–2.5 h more, the reaction was quenched and worked-up as described in general procedure A. The crude product was purified by silica-gel column chromatography (ethyl acetate/hexane = 1:15 then 1:3) to afford *syn*- and *anti*-**3i–p** as pure stereoisomers, or as a mixture of diastereomers.

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Keywords: aldol reaction · conjugate reduction · organocatalysis · tandem reaction · Wittig reactions

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