JOC The Journal of Organic Chemistry

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Diastereoselectivity Switch in Cooperatively Catalyzed Three-Component Reactions of an Aryldiazoacetate, an Alcohol, and a β,γ-Unsaturated α-Keto Ester

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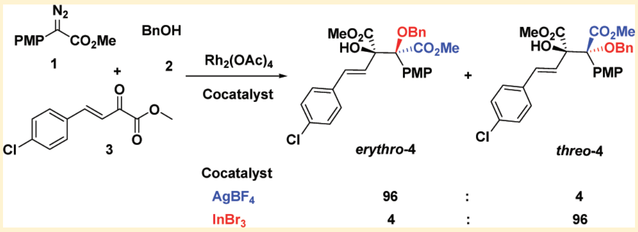
S Supporting Information

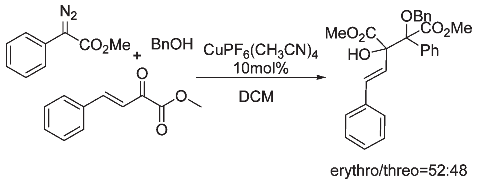
ABSTRACT: A highly switchable,diastereoselectivity-controlled, three-component reaction of aryldiazoacetate, alcohol,and β,γ-unsaturated α-keto esters has been achieved through a dual-metal cooperative catalysis strategy.



Efficient construction of acyclic molecules bearing multiple stereogenic centers with complete stereoselective control in a designed way continues a great challenge in modern organic synthesis. The reversal of absolute configuration to the opposite enantiomer can be easily achieved by switching the chiral catalyst to its enantiomer in asymmetric catalysis. However, development of a diastereoselective switchable strategy providing high-level control of diastereoselectivity of the newly formed stereocenters for each diastereomer has been much more challenging. One fundamental strategy is to utilize substrate stereochemistry to control the introduction of new stereogenic centers through "chelation and nonchelation" modes to control the resulting diastereoselectivity.[1] In some cases, reversal of the diastereoselectivity can be accomplished by adjusting the size of the protecting group in substrates.[2] There have been only a few successful examples documented in tuning of diastereoselectivity while using the same achiral substrates in the switchable controlling process.[3,4] For example, Hou and co-workers reported a strategy of modifying the electronic properties of a chiral phosphine ligand in order to achieve a dramatic anti/syn diastereoselectivity switch in a Mannich reaction.[4a]

In comparison to the traditional chemistry of two-component reactions, multicomponent reactions (MCRs) offer great advantages in multibond formation within one synthetic operation from simple substrates to construct complex molecules that contain multiple stereogenic centers.[5,6] Stereoselective control in the MCRs remains a challenge in the multibond formation process. Recently, as an effective strategy to enhance reaction selectivity and reactivity, cooperative catalysis has been extensively reported in two-component reactions with improved synthetic efficiency.[7,8] We have applied such a strategy to effectively control enantioselectivity in multicomponent reactions based on an oxonium ylide trapping process.[9] We report here a highly controlled diastereoselectivity switch in a dual-metal cocatalyzed three-component reaction. The flexibility during the assembly process of the multiple components is found to be advantageous in the controllable tuning of the diastereoselectivity.

Scheme 1. Three-Component Reaction of Phenyldiazoacetate, Alcohol and β,γ-Unsaturated α-Keto Esters

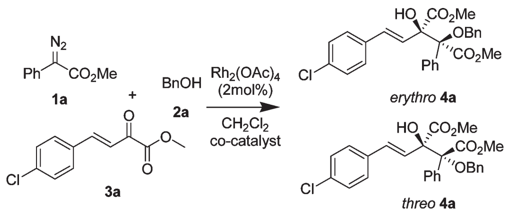


In our previous work, we reported a copper(I)-catalyzed three-component reaction of methyl phenyldiazoacetate with benzyl alcohol and a β,γ-unsaturated α-keto ester (Scheme 1).[10] The reaction efficiently afforded an α,β-dihydroxy acid derivative in moderate yield, however, with very poor diastereoselectivity (dr = 52:48). Inspired by recent successes in applying a cooperative catalysis strategy to enhance reaction selectivity, we envisioned that it should be possible to enhance the diastereoselectivity of the reaction by appropriately choosing a suitable and compatible cocatalytic system.

Initially, we began to investigate the reaction of methyl phenyldiazoacetate (**1a**), benzyl alcohol (**2a**) and the β,γ-unsaturated α-keto ester **3a** in the presence of $Rh_2(OAc)_4$ catalyst alone. The desired three-component product **4a** was obtained in low yield with good diastereoselectivity, favoring *erythro*-**4a** (Table 1, entry 1). We started to screen cocatalysts to activate the keto

Received: December 12, 2010
Published: June 13, 2011

Table 1. Screening of Cocatalysts[a]



entry	cocat. (10 mol %)	yield (%)[b,c]	erythro:threo dr[d]
1		37	90:10
2	AgOTf	32	93:7
3	$AgSbF_6$	38	96:4
4	$AgBF_4$	48	96:4
5[e]	$AgBF_4$	52	96:4
6	$Sc(OTf)_3$	27	80:20
7	$Yb(OTf)_3$	28	78:22
8	$Sn(OTf)_2$	56	45:55
9	$In(OTf)_3$	42	37:63
10	$Zn(OTf)_2$	46	33:67
11	$InBr_3$	60	10:90
12[f]	$InBr_3$	76	4:96

[a] Unless otherwise noted, the reaction was carried out by addition of **1a** (0.24 mmol, 1.2 equiv) in CH_2Cl_2 (1 mL) to a mixture of **2a** (0.24 mmol, 1.2 equiv), **3a** (0.2 mmol, 1.0 equiv), and 4 Å MS (120 mg) in the presence of 2 mol % of $Rh_2(OAc)_4$ and 10 mol % of cocatalyst in 4 mL of CH_2Cl_2 for 1 h at room temperature. [b] Isolated yield. [c] Separated as a mixture of erythro and threo isomers. [d] The ratio was determined by ^1H NMR spectroscopy of the crude reaction mixture. [e] 1.5 equiv of **1a** was used. [f] 20 mol % of $InBr_3$ was used.

ester, hoping to enhance the diastereoselectivity and product yield. We were pleased to find that silver complexes were the cocatalyst of choice to give enhanced diastereoselectivity of *erythro*-**4a** (Table 1, entries 2−5). $AgBF_4$ was found to be the best cocatalyst to afford *erythro*-**4a** in 48% isolated yield and excellent diastereoselectivity (dr = 96:4) (Table 1, entry 4). A control reaction was conducted, and the use of $AgBF_4$ catalyst alone gave less 5% of the coupling product. The isolated product yield was slightly improved to 52% when the amount of component **1a** was increased from 1.2 to 1.5 equiv (Table 1, entry 5). During the process of screening the cocatalyst, we found that some Lewis acids with better chelation capability ruined the diastereoselectivity (Table 1, entries 6−8). For example, cocatalyst $Sn(OTf)_2$ gave product **4a** in 56% yield with dr = 45:55 (Table 1, entry 8), indicating that Lewis acids with better chelation capability favored the formation of *threo*-**4**. We thought there might be an opportunity to completely change the diastereoselectivity to *threo*-**4a** through chelation control. Additional Lewis acids were tested, and $In(OTf)_3$ and $Zn(OTf)_2$ were found to afford *threo*-**4a** with about dr = 2:1 favoring *threo*-**4a** (Table 1, entries 9 and 10). Gratifyingly, $InBr_3$ led to *threo*-**4a** in 60% yield with dr = 90:10 (Table 1, entry 11), and by increasing the catalyst loading to 20 mol %, we were able to get *threo*-**4a** in 76% yield with dr = 96:4 (Table 1, entry 12).

The reaction scope was evaluated under the optimized conditions. A variety of β,γ-unsaturated α-keto esters, alcohols, and diazo compounds were investigated under these reaction conditions, using $AgBF_4$ or $InBr_3$ as a cocatalyst to selectively give each desired diastereomer, *erythro*-**4** or *threo*-**4**, respectively. The results are shown in Table 2. In general, the reaction has good tolerance to substitution on the aromatic rings of the substrates, as good yields and excellent diastereoselectivities in a controlled switchable manner were obtained for those representative substrates. One exception was the reaction with *p*-methoxybenzyl alcohol. While a good yield and excellent dr value of *erythro*-**4** were obtained with $AgBF_4$, the switch to its diastereomer with a change to the $InBr_3$ cocatalyst failed to produce *threo*-**4**. It is likely that the *p*-methoxybenzyl alcohol has better coordination ability, which inhibits the catalytic activity of $InBr_3$. In fact, we observed a dramatic color change when mixing $InBr_3$ with the alcohol in solution, indicating the coordination of the alcohol to $InBr_3$. The stereochemistry of *erythro*-**4b** was confirmed by a single-crystal X-ray analysis (Figure 1).

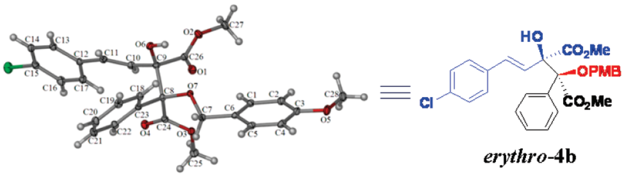
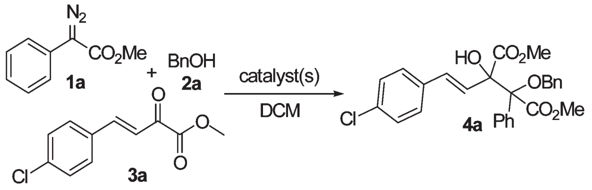


Figure 1. X-ray crystal structure of *erythro*-**4b**.

Scheme 2. $Rh_2(4R\text{-MEOX})_4$-Catalyzed Three-Component Reaction of 1a, 2a, and 3a

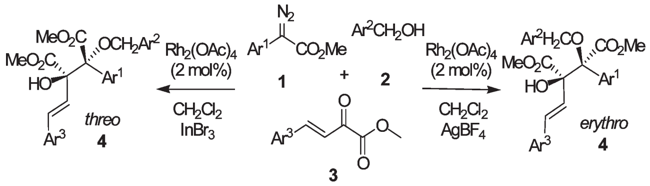


	yield,	*erythro (% ee) : threo (% ee)*
$Rh_2(4R\text{-MEOX})_4$:	30%,	89 (0) : 11 (0)
$Rh_2(4R\text{-MEOX})_4/AgBF_4$:	29%,	92 (0) : 8 (0)
$Rh_2(4R\text{-MEOX})_4/InBr_3$:	26%,	14 (12) : 86 (11)

To get insight into the reaction mechanism, we have conducted the reaction in the presence of a chiral dirhodium catalyst. Thus, Doyle's $Rh_2(4R\text{-MEOX})_4$[11] was used to replace $Rh_2(OAc)_4$ in the three-component reaction of **1a**, **2a**, and **3a** (Scheme 2). Doyle's catalyst was found to be less effective than $Rh_2(OAc)_4$, as a lower yield was obtained. Similarly low yields were obtained in the presence of the cocatalyst $AgBF_4$ or $InBr_3$. However, in the presence of $InBr_3$ cocatalyst, we still observed the reversed diastereoselectivity favoring *threo*-**4a**. In addition, we observed detectable enantioselectivity in this case, indicating that a rhodium-associated oxonium ylide might be involved in a transition state.

The reaction mechanism that accounts for the diastereoselectivity switch in the three-component reaction is not yet clear. However, plausible transition states are proposed in order to explain the observation (Scheme 3). $Rh_2(OAc)_4$ plays a role in the generation of the active metal-associated oxonium ylide **5** from the diazo compound and alcohol. In the presence of $Rh_2(OAc)_4$ alone or in the presence of $AgBF_4$, the reaction proceeds through a conformation, as in intermediate **6**, leading to *erythro*-**4** as a major isomer. In the case of $InBr_3$, which has a high

Table 2. Three-Component Switchable Diastereoselectivity-Controlled Reactions of Methyl Aryldiazoacetate with Alcohol and β,γ-Unsaturated α-Keto Esters



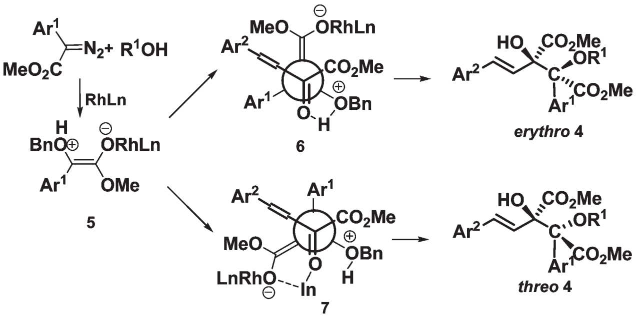
entry	Ar^1/Ar^2/Ar^3	4	AgBF$_4$[a] yield (%)[c]	AgBF$_4$[a] erythro:threo dr[d]	InBr$_3$[b] yield (%)[c]	InBr$_3$[b] erythro:threo dr[d]
1	Ph/Ph/4-ClPh	**4a**	52	96:4	76	4:96
2	Ph/4-MeOPh/4-ClPh	**4b**	54	>96:4	<5	N.D.
3	Ph/4-ClPh/4-ClPh	**4c**	45	96:4	71	5:95
4	Ph/4-BrPh/4-ClPh	**4d**	46	95:5	70	4:96
5	4-BrPh/Ph/4-ClPh	**4e**	37	96:4	58	<4:96
6	4-OMePh/Ph/4-ClPh	**4f**	70	96:4	74	<4:96
7	4-OMePh/Ph/Ph	**4g**	66	96:4	65	4:96
8	4-OMePh/Ph/4-FPh	**4h**	50	96:4	70	4:96
9	4-OMePh/Ph/4-MePh	**4i**	60	>96:4	76	5:95
10	4-OMePh/Ph/3-MePh	**4j**	74	>96:4	61	4:96
11	4-OMePh/Ph/3-BrPh	**4k**	64	>96:4	61	9:91

[a] All reactions were carried out by addition of **1** (0.30 mmol, 1.5 equiv) in CH_2Cl_2 (1 mL) via a syringe pump to a mixture of 2 mol % of $Rh_2(OAc)_4$, **2** (0.24 mmol, 1.2 equiv), **3** (0.2 mmol, 1.0 equiv), 4 Å MS (120 mg), and 10 mol % of $AgBF_4$ in 4 mL of CH_2Cl_2 for 1 h at room temperature. [b] All reactions were carried out by addition of **1** (0.24 mmol, 1.2 equiv) in CH_2Cl_2 (1 mL) via a syringe pump to a mixture of 2 mol % of $Rh_2(OAc)_4$, **2** (0.24 mmol, 1.2 equiv), **3** (0.2 mmol, 1.0 equiv), 4 Å MS (120 mg), and 20 mol % of $InBr_3$ in 4 mL of CH_2Cl_2 for 1 h at room temperature. [c] Isolated yield of a mixture of erythro and threo isomers. [d] The ratio was determined by ^{1}H NMR spectroscopy of the crude reaction mixture.

coordination ability, the ester group from the diazo compound and the carbonyl group from the keto ester are brought close together via coordination with $InBr_3$ to form a transition state, such as **7**, leading to the formation of *threo*-**4**. The requisite coordination of the diazo compound and the keto ester with $InBr_3$, leading to transition state **7**, could be easily achieved prior to the rhodium-catalyzed oxonium ylide formation with the alcohol component. Thus, it is advantageous for controlling the diastereoselectivity in the three-component reaction.

In conclusion, a highly switchable, diastereoselectivity-controlled, three-component reaction of an aryl diazoacetate, an alcohol, and a β,γ-unsaturated α-keto ester has been achieved. A dual-metal cooperative catalysis strategy was applied to control each desired diastereomer. A similar strategy may be applied in other MCRs to control the reaction selectivity.

Scheme 3. Proposed Reaction Pathway for Diastereoselectivity Switch



■ EXPERIMENTAL SECTION

General Procedure for Diastereoselectivity Switch in Co-operatively Catalyzed Three-Component Reactions of an Aryldiazoacetate, an Alcohol, and a β,γ-Unsaturated α-Keto Ester. A 10 mL dry reaction vial was charged with alcohol **2** (0.24 mmol), β,γ-unsaturated α-keto ester **3** (0.2 mmol), $Rh_2(OAc)_4$ catalyst (2 mol %), a cocatalyst (10 mol % $AgBF_4$ or 20 mol % $InBr_3$), and 4 Å MS (120 mg), and 4 mL of CH_2Cl_2 was added to the vial. The resulting mixture was stirred at ambient temperature for 10 min. To the above mixture was added methyl aryldiazoacetate **1** (0.3 mmol for the $AgBF_4$ system, 0.24 mmol for the $InBr_3$ system) in 1 mL of CH_2Cl_2 over 1 h via a syringe pump. After completion of the addition, the reaction mixture was stirred for an additional 30 min. The reaction mixture was filtered and concentrated under reduced pressure. The crude product was subjected to ^{1}H NMR analysis for the determination of the diastereoselectivity. The crude product was purified by flash chromatography on silica gel (eluent EtOAc/light petroleum, 1/20−1/10) to give **4** as a mixture.

(2R,3S*,E)-Dimethyl 2-(4-Chlorostyryl)-3-(benzyloxy)-2-hydroxy-3-phenylsuccinate (erythro-**4a**).* Yield: 52%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.64 (m, 2H), 7.36−7.30 (m, 12H), 6.73 (d, J = 15.9 Hz, 1H), 6.60 (d, J = 15.9 Hz, 1H), 4.73 (d, J = 12.1 Hz, 1H), 4.50 (d, J = 12.1 Hz, 1H), 4.14 (s, 1H), 3.80 (s, 3H), 3.68 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.1, 170.1, 138.4, 135.2, 133.3, 133.2, 130.1, 128.7, 128.5, 128.5, 128.2, 127.9, 127.5, 127.2, 126.5, 126.1, 89.5, 82.1, 68.4, 53.0, 52.1. HRMS (ESI): m/z calcd for $C_{27}H_{25}ClNaO_6$ $(M + Na)^+$ 503.1232, found 503.1235.

(2R,3R*,E)-Dimethyl 2-(4-Chlorostyryl)-3-(benzyloxy)-2-hydroxy-3-phenylsuccinate (threo-**4a**).* Yield: 76%. ^{1}H NMR (500 MHz,

$CDCl_3$): δ 7.49−7.47 (m, 2H), 7.39−7.36 (m, 4H), 7.30−7.28 (m, 4H), 7.21 (d, J = 8.5 Hz, 2H), 7.17−7.15 (d, J = 8.5 Hz, 2H), 6.47 (d, J = 15.9 Hz, 1H), 6.43 (d, J = 15.9 Hz, 1H), 4.74 (d, J = 12.3 Hz, 1H), 4.60 (d, J = 12.3 Hz, 1H), 4.40 (s, 1H), 3.82 (s, 3H), 3.77 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.3, 170.7, 138.5, 135.0, 133.6, 133.2, 130.5, 129.0, 128.5, 128.5, 128.2, 127.8, 127.2, 127.1, 126.5, 126.2, 90.1, 81.2, 68.5, 53.2, 52.3. HRMS (ESI): m/z calcd for $C_{27}H_{25}ClNaO_6$ $(M + Na)^+$ 503.1232, found 503.1222.

■ ASSOCIATED CONTENT

Supporting Information. Text and figures giving experimental procedures, characterization data, ^1H and ^{13}C NMR spectra, and HPLC of new compounds and tables, a figure, and a CIF file giving crystallographic data for *erythro*-**4b**. This material is available free of charge via the Internet at http://pubs.acs.org.

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■ ACKNOWLEDGMENT

We are grateful for financial support from the National Natural Science Foundation of China (Grant Nos. 20772033 and 20932088), the Shanghai Municipal Education Commission (Grant No. 09ZZ45), and the Science and Technology Commission of Shanghai Municipality (Grant Nos. 09JC1404900 and 10XD1401700).

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