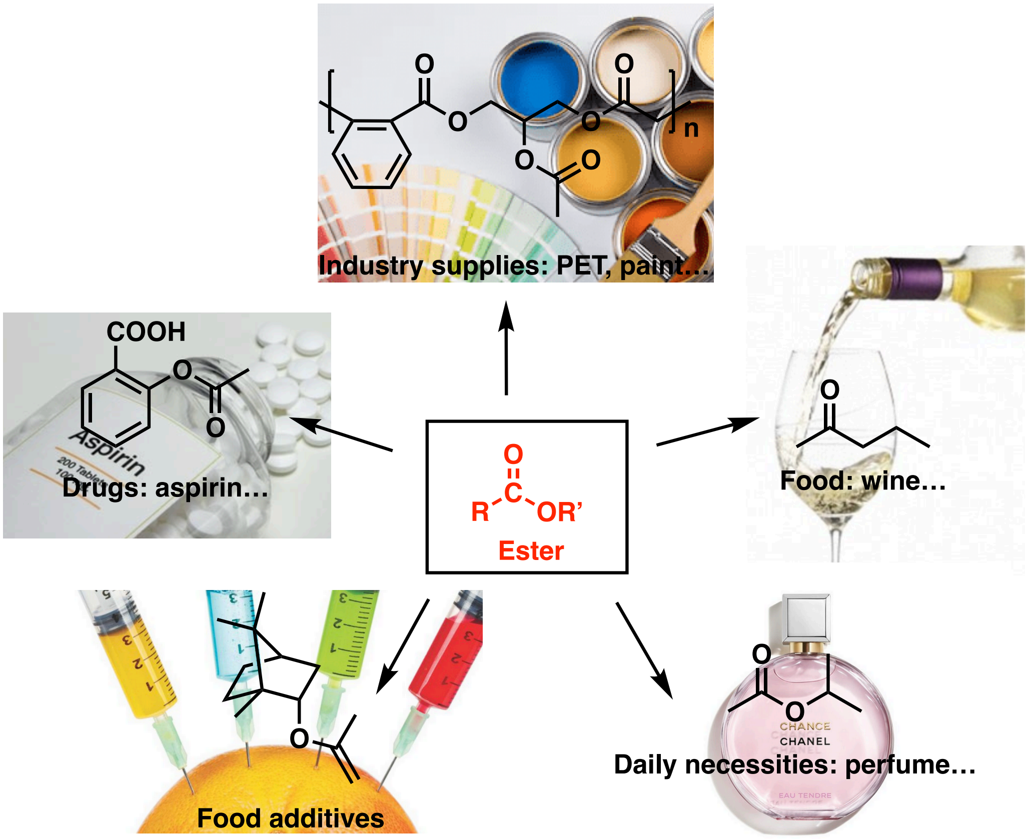
Contents

1. Introduction

1-1. Basic Information About Esters.

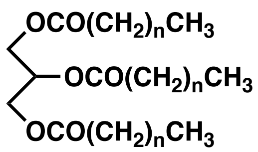
-What are the esters used for?



-Where are the esters come from?

(1) From nature:

⇨ Fats and oil are formed known as triglycerides
Naturally occuring in plants…



triglycerides

(2) From artficial:

⇨ Esterification of carboxylic acids with alcohols

$$RCOOH + R^1OH \rightleftharpoons RCOOR^1 + H_2O$$

⇨ Esterification of carboxylic acids with epoxides

$$RCOOH + R^1CHCH_2O \longrightarrow RCOOCH_2CH(OH)R^2$$

⇨ Alcoholysis of acyl chlorides and acid anhydrides

$$RCOCl + R^2OH \longrightarrow RCOOR^2 + HCl$$

⇨ Transesterification

$$RCOOR^1 + R^2OH \longrightarrow RCOOR^2 + R^1OH$$

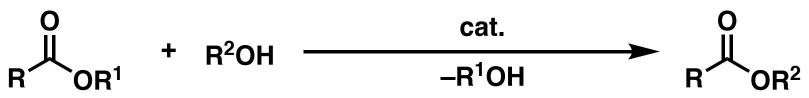
⇨ And so on…

1. Introduction

1-2. Basic Information About Transesterification

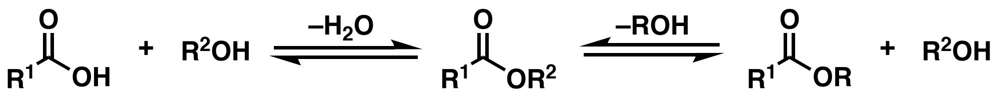
-What is transesterification?

Transesterification is the process of exchanging the organic group R^1 of an ester with the organic group R^2 of an alcohol.



General transesterification

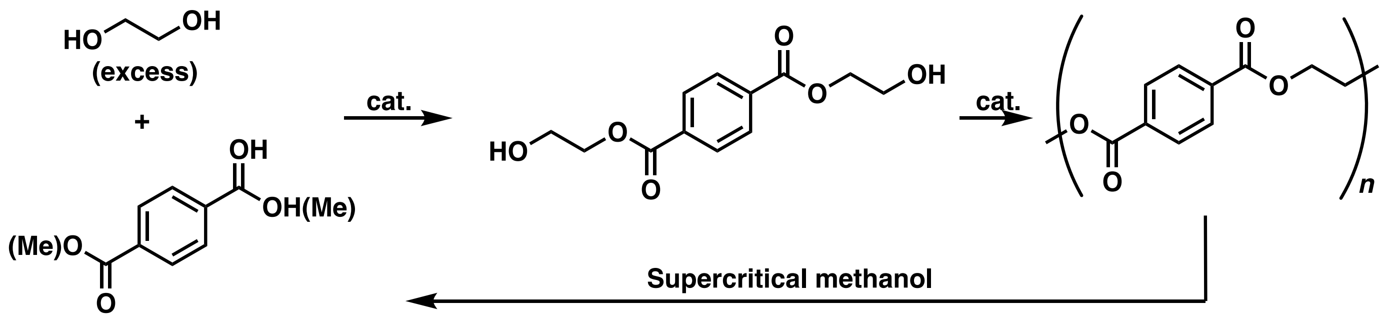
-What is the difference between direct dehydrative condensation and transesterification?



- Direct dehydrative condensation
  - Byproduct is H_2O only.
  - Carboxylic acid is low solubility.
  - Carboxylic acid can play as catalyst.
- Transesterification
  - If methyl ester is used, byproduct is MeOH only.
  - Carboxylic ester is high solubility.
  - Base catalyst can be used.

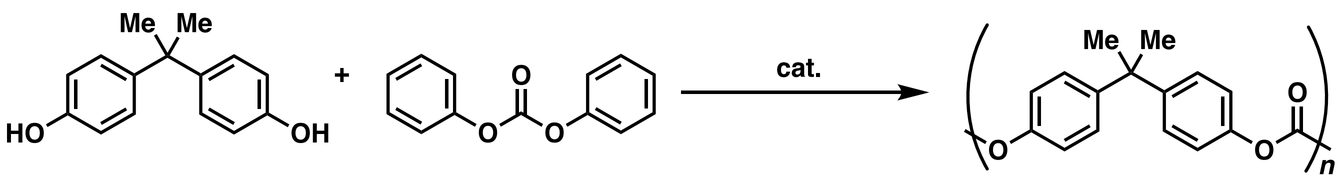
- Industrial use of carboxylate ester, which are produced by transesterification

- PET (textile fibers, films, bottle, resins, plastics……)

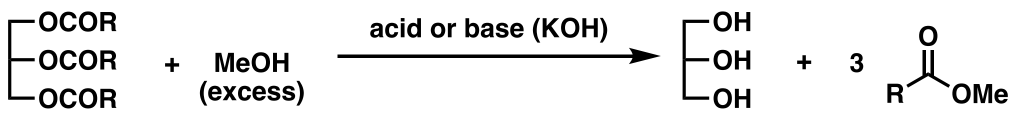


cat.: Pb(II)$(OAc)_2$, Pb(IV)$(OAc)_4$, $Zn(OAc)_2$, $Mg(OAc)_2$, $Ca(OAc)_2$, $Co(OAc)_2$, $Cd(OAc)_2$, Sb_2O_3, Ge_2O_3, $Ti(OR)_4$

- Polycarbonate



- Biodiesel, Oils, Fats, Soaps

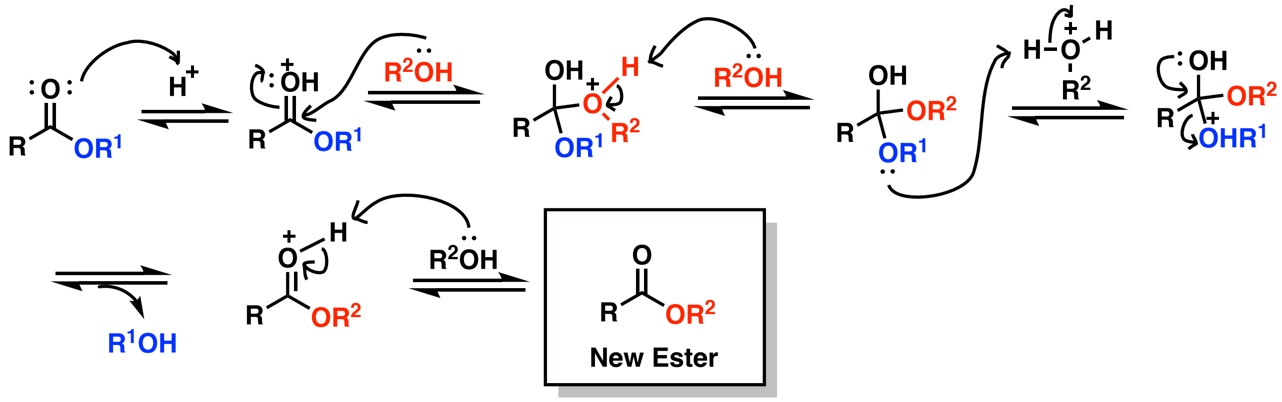


R = long alkyl chain

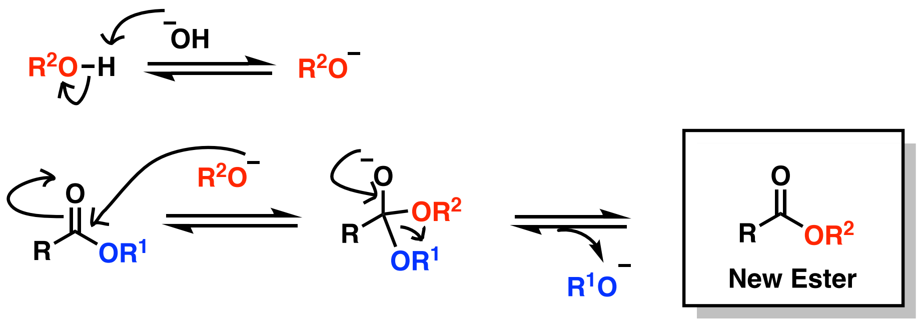
2. Mechanism and Characteristics

2-1. Mechanism

-Acid-Catalyzed Transesterification Mechanism



-Base-Catalyzed Transesterification Mechanism



Catalyst

- **Acid Catalysts**: HCl, H_2SO_4, HI, HF, HBF_4, $HClO_4$, Metal Alkoxide……
- **Base Catalysts**: Et_3N, DBU……
- **N-Heterocyclic Carbene Catalyst**: IMes, ICy……

2-2. Characteristics

-Transesterification can be catalyzed by both acid and base catalysts.

-The transesterification reaction is an equilibrium reaction, which requires the addition of molecular sieves or reflux to remove the produced alcohol in order to shift the equilibrium.

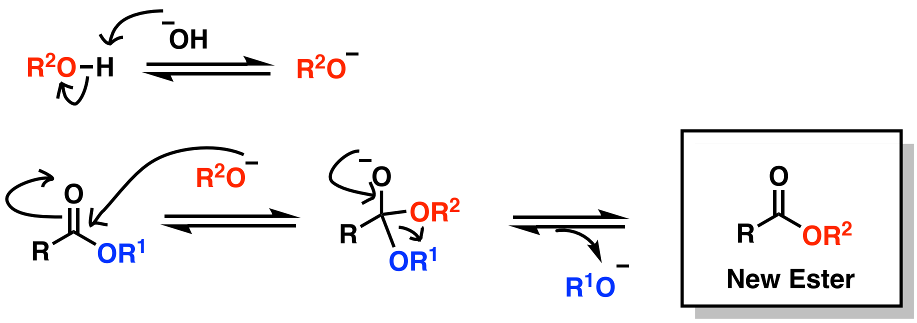
-Transesterification can be cinducted under anhydrous conditions, to allow employment of moisture-sensitive materials.

-Transesterification is applicable not only to the pure organic synthesis but also to polymerization.

3. Catalysts

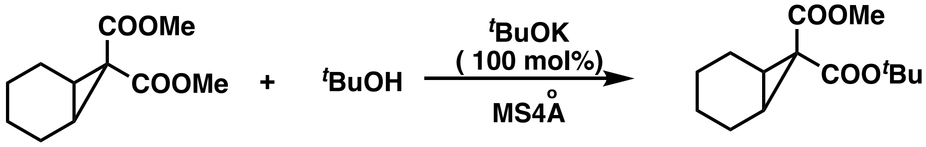
3-1. Base Catalysts

3-1-1. Homogeneous Catalysts



General Base-Catalyzed Transesterification Mechanism

^{t}BuOK [1)]



- Full conversion with MS4Å,
- Only 80% conversion without MS4Å

liq.NH_3 [2)]

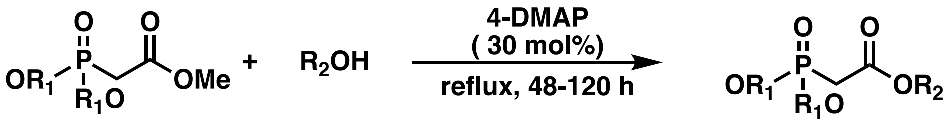
ArCOOPh + ROK →(liq.NH_3) ArCOOR

Ar = Ph, *o*-MeC_6H_5, *m*-ClC_6H_4 ...

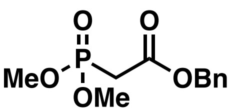
R = *t*-Bu, *t*-amyl, *iso*-Bu

- 10 examples
- 76~92% Yield

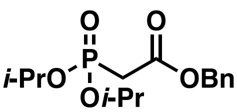
4-DMAP [3)]



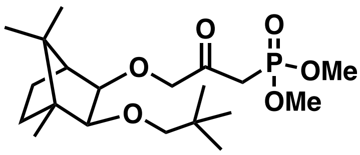
Substrate, Time, Yield



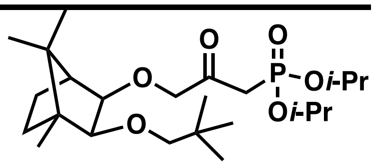
48 h, 48%



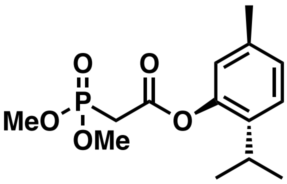
48 h, 86%



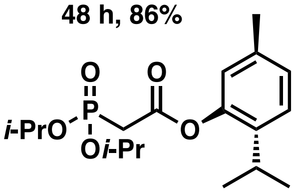
72 h, 74%



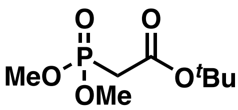
120 h, 91%



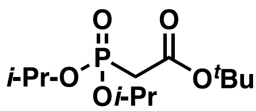
72 h, 84%



72 h, 96%



120 h, N.R



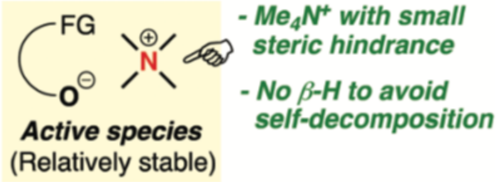
120 h, N.R

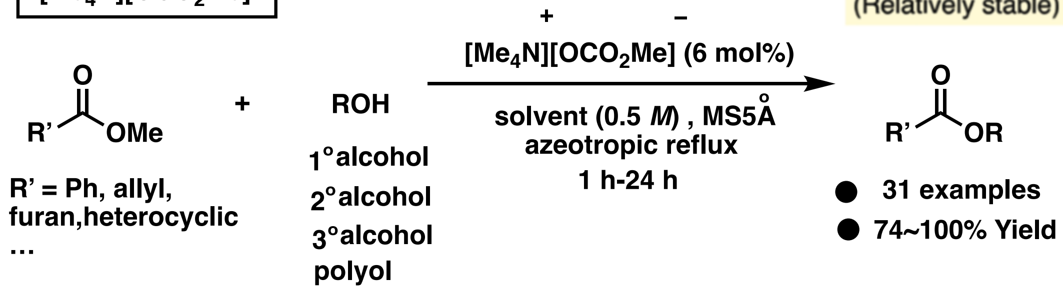
3. Catalysts

3-1. Base Catalysts

3-1-1. Homogeneous Catalysts

[Me$_4$N]$^+$[OCO$_2$Me]$^-$ [4)]





2a: [Me(n-octyl)$_3$P]$^+$[OCO$_2$Me]$^-$

3a: [Me(n-octyl)$_3$N]$^+$[OCO$_2$Me]$^-$

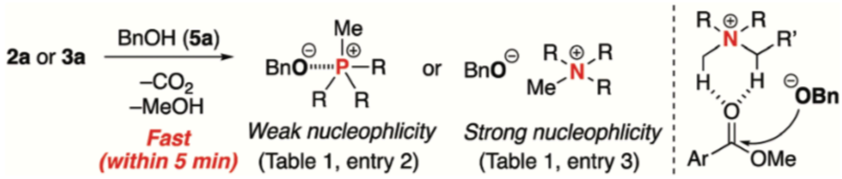
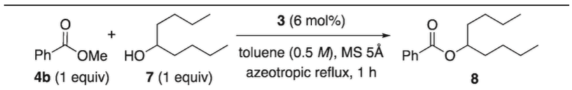


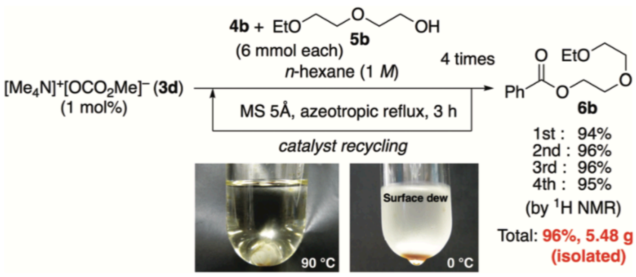
Fig. 2 Active species *in situ* from **2a** or **3a**.

Table 2 Screening of ammonium salt catalysts **3**[a]



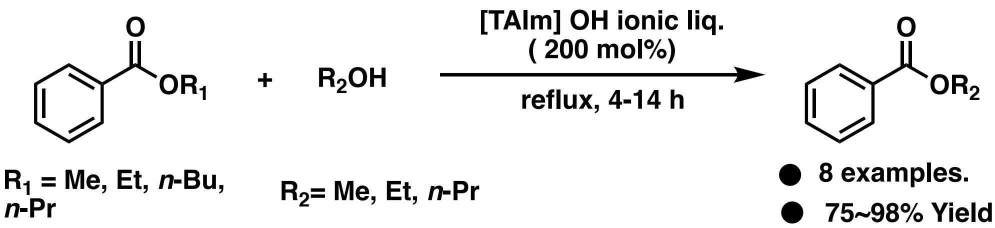
Entry	Catalyst	Yield (%)
1	[Me(n-octyl)$_3$N]$^+$[OCO$_2$Me]$^-$ (**3a**)	26
2	[Me$_2$(n-octyl)$_2$N]$^+$[OCO$_2$Me]$^-$ (**3b**)	57
3	[Me$_3$(n-octyl)N]$^+$[OCO$_2$Me]$^-$ (**3c**)	64
4	[Me$_4$N]$^+$[OCO$_2$Me]$^-$ (**3d**)	85
5	[Et$_4$N]$^+$[OCO$_2$Me]$^-$ (**3e**)	0
6	[Me$_4$N]$^+$[OCO$_2$H]$^-$ (**3f**)	6
7	[Me$_4$N]$^+$[OH]$^-$ (**3g**)	17
8	[Me$_4$N]$^+$[Cl]$^-$ (**3h**)	0

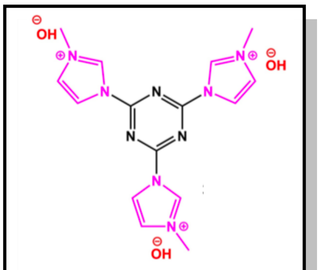
[a] The reaction was carried out with **4b** (2 mmol), 7 (2 mmol), and catalyst (6 mol%) in toluene (bp. 110 °C) at 140 °C (bath temperature) for 1 h. The same reactor system was used as in Table 1.



Scheme 1 Recovery and reuse of the catalyst in the gram scale synthesis of **6b**.

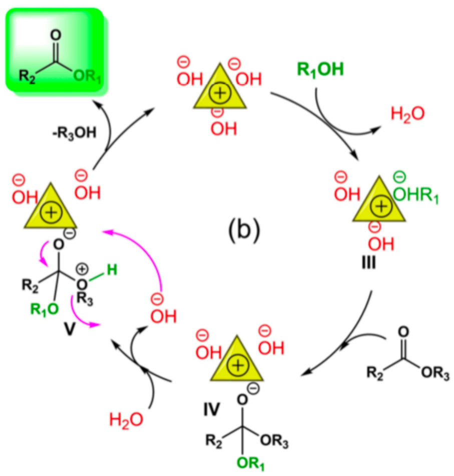
[TAIm] OH [5)]

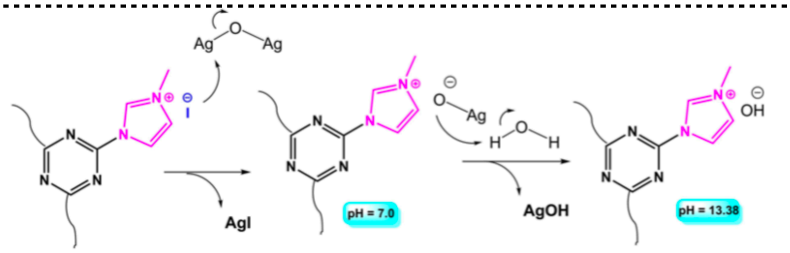




Catalyst

Proposed Mechnism





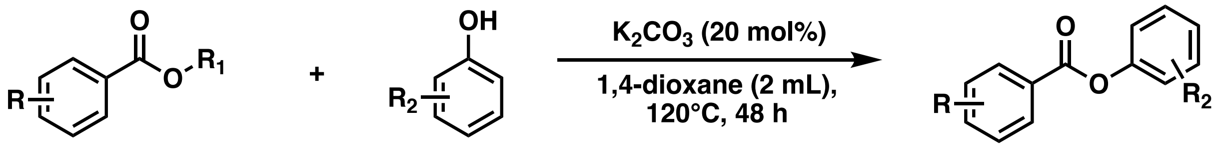
Scheme 3. A mechanistic view for Hofmann elimination during the preparation of [TAIm]OH ionic liquid.

3. Catalysts

3-1. Base Catalysts

3-1-2. Heterogeneous Catalysts

K_2CO_3 [6)]



R_1 = Me, Ph, *o*-MeC_6H_5, *p*-$NO_2C_6H_5$, *N*-hetero…

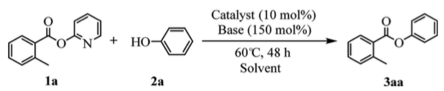
R_1 = C_6F_5, *N*-hetero…

R_2= Cl, NO_2, OMe, Ph

- 15 examples.
- 37~84% Yield

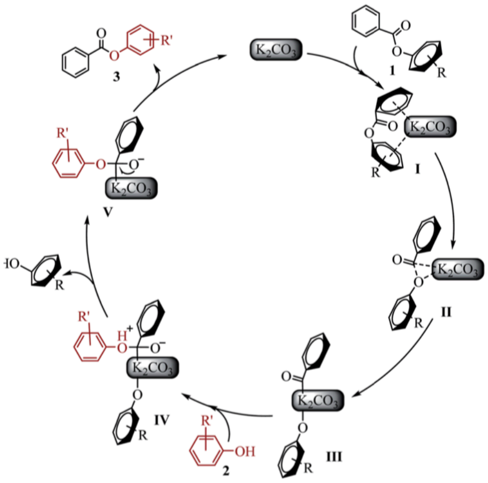
Screening of different reaction parameters

Table 1 Screening of different reaction parameters[a]

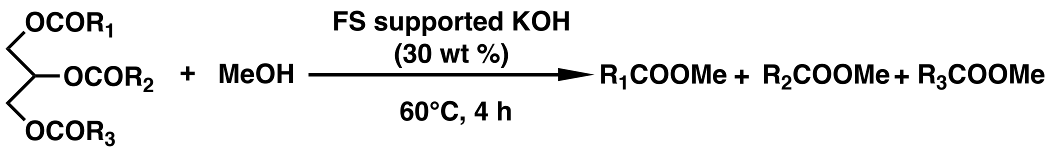


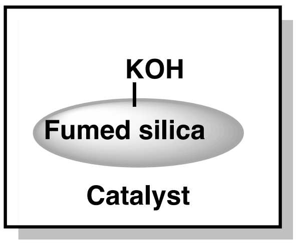
Entry	Catalyst	Base	Solvent	Yield[b] (%)
1	Pd/γ-Al_2O_3	K_2CO_3	1,4-Dioxane	99
2	K_2CO_3	—	1,4-Dioxane	Quant
3	K_3PO_4	—	1,4-Dioxane	81
4	KO^tBu	—	1,4-Dioxane	79
5	KOH	—	1,4-Dioxane	70
6	Cs_2CO_3	—	1,4-Dioxane	93
7	NaOH	—	1,4-Dioxane	NP
8	Li_2CO_3	—	1,4-Dioxane	NP
9	$Ca(OH)_2$	—	1,4-Dioxane	NP
10	NEt_3	—	1,4-Dioxane	NP
11	NaOH	—	DMSO	48

Proposed Mechnism

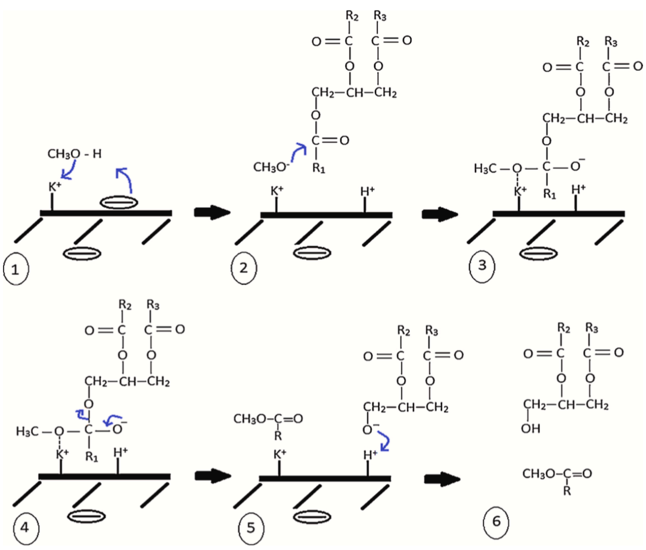


FS supported KOH [7)]

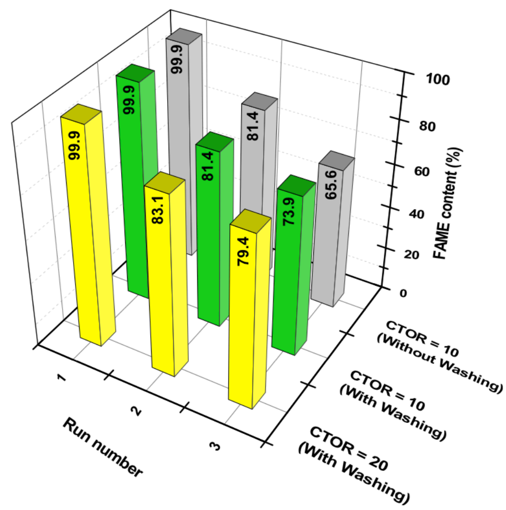




Proposed Mechnism



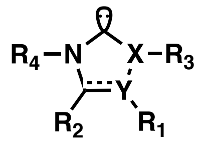
Reuse of the catalyst



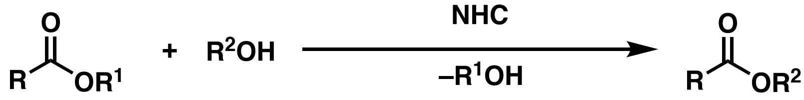
3. Catalysts

3-2. *N*-Heterocyclic Carbene Catalysts

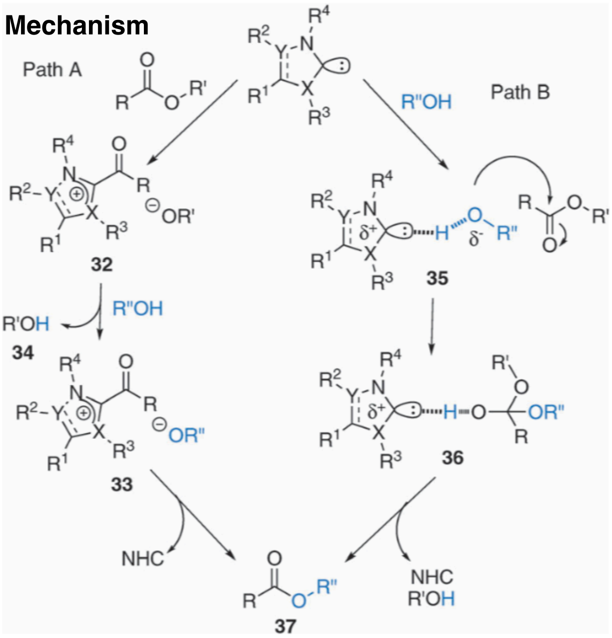
3-2-1. Homogeneous Catalysts

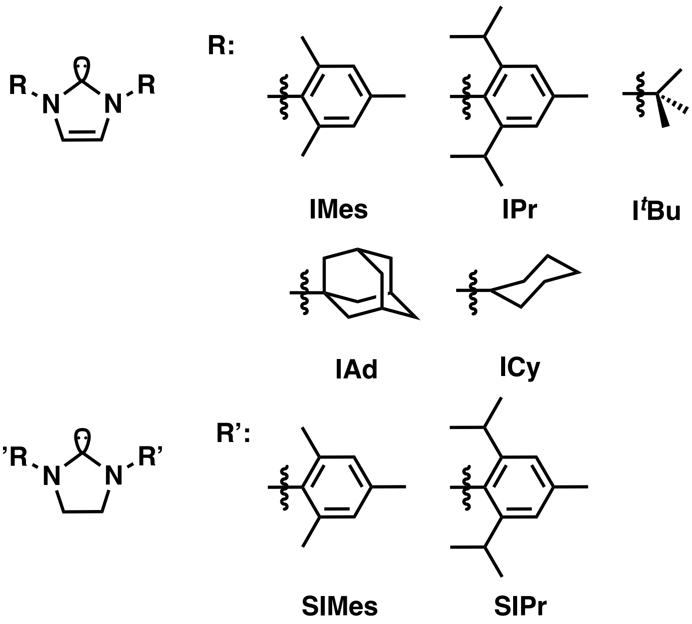


N-Heterocyclic Carbene Catalyst

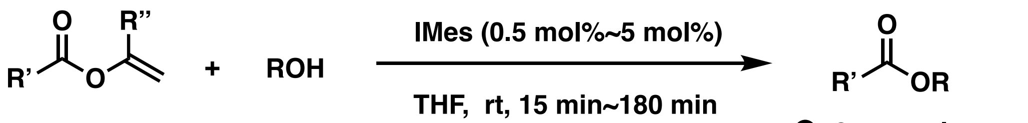


Various *N*-Heterocyclic Carbenes





IMes 8)

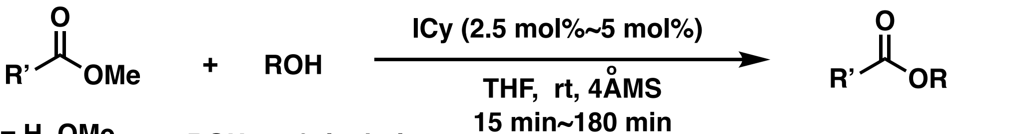


R' = Me, Ph, CH_2
R" = H, Me

2°alcohol

- 8 examples
- 83~100% Yield

ICy 8)

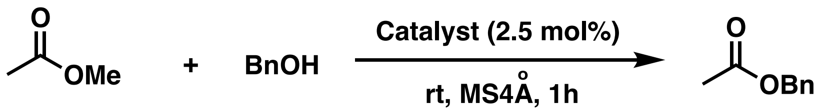


R' = H, OMe, thiophene...

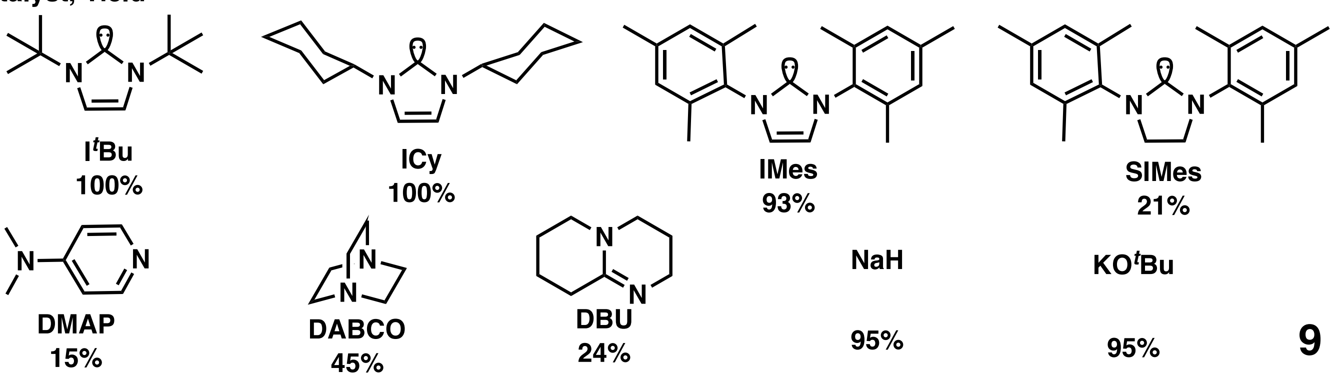
ROH = 2°alcohol
3°alcohol

- 12 examples
- 89~99% Yield

others nucleophilic catalysts 8)



Catalyst, Yield



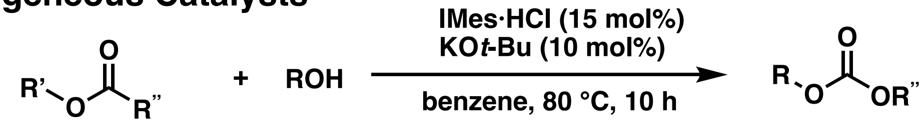
NaH 95%

KOtBu 95%

3-2. *N*-Heterocyclic Carbene Catalysts

3-2-2. Heterogeneous Catalysts

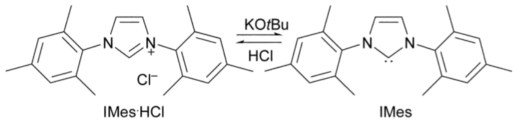
IMes·HCl [9)]



R' = CH_3, CH_2CH_3, allyl
R'' = H, CH_3, CH_2Br

1°alcohol

- 21 examples
- 17~95% Yield



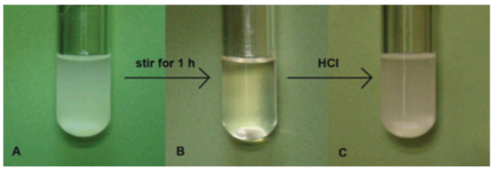
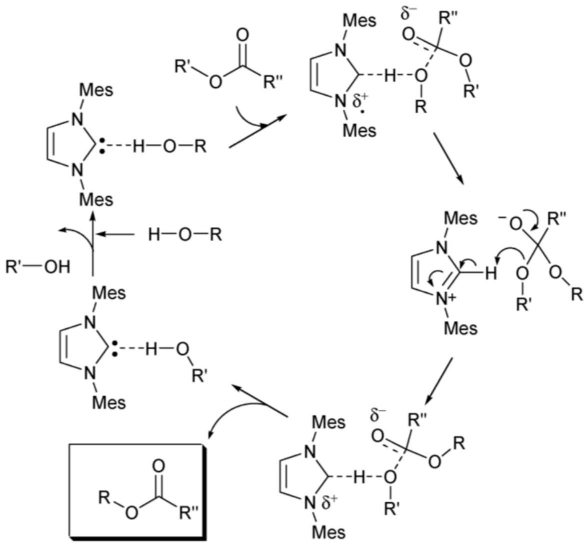


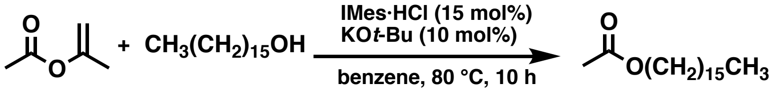
Fig. 1 A: 0.2 mmol IMes·HCl and 0.2 mmol KO*t*Bu in 2.5 mL benzene. B: after being stirred for 1 h. C: after addition of 0.2 mmol HCl (1 M in Et_2O).

Proposed Mechanism



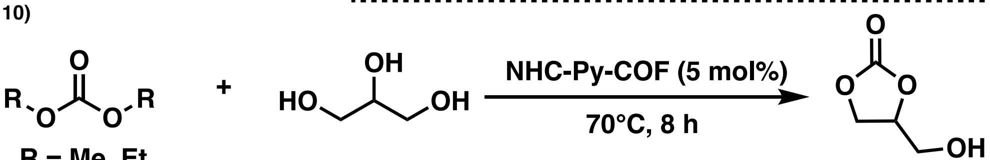
Scheme 2 Proposed reaction mechanism.

Reuse of the catalyst



Run	Isolated Yield (%)
1	75
2	80
3	98
4	80
5	84
6	85
7	77

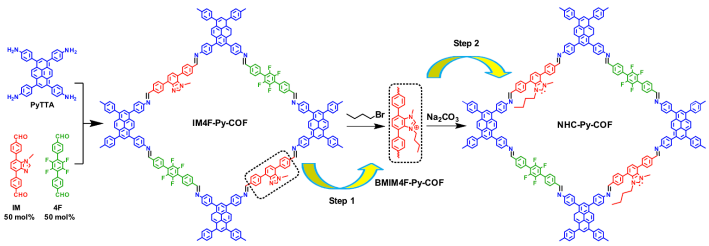
NHC-Py-COF [10)]



R = Me, Et

- 2 examples
- 96% Yield

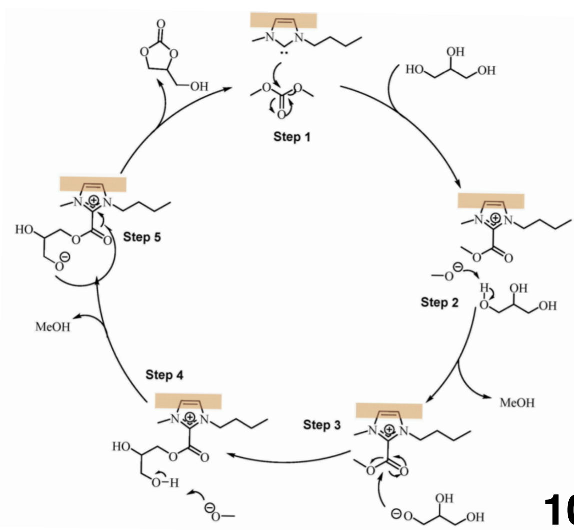
Synthesis of NHC-Py-COF



Entry	Catalyst	Substrate (R=)	Conversion [b] (%)	Yield [b] (%)
1	Blank	CH_3	—	—
2	NHC-Py-COF	CH_3	99	96
3	NHC-Py-COF	CH_2CH_3	96	96
4	NHC-[BMIM]Br	CH_3	99	99
5	[BMIM]Br	CH_3	—	—
6	IM4F-Py-COF	CH_3	—	—
7	4F-Py-COF	CH_3	—	—
8 [c]	NHC-Py-COF	CH_3	99	95
9 [d]	NHC-Py-COF	CH_3	98	95
10 [e]	NHC-Py-COF	CH_3	98	94
11 [f]	NHC-Py-COF	CH_3	97	94
12 [g]	NHC-Py-COF	CH_3	95	93

[a] Reaction condition: catalyst 5 mol%, glycerol (2.5 mmol), dialkyl carbonate (7.5 mmol), 70 °C, 8h. [b] Determined by GC and confirmed by ^1H NMR analysis. [c–g] Cycle from one to five times, respectively.

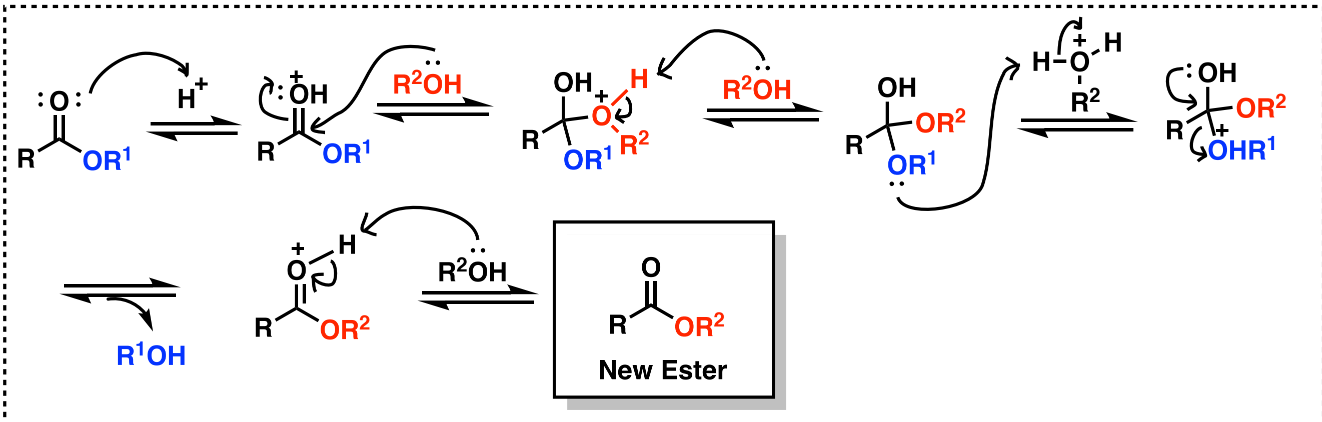
Proposed Mechanism



3. Catalysts

3-3. Brønsted Acid

3-3-1. Homogeneous Catalysts



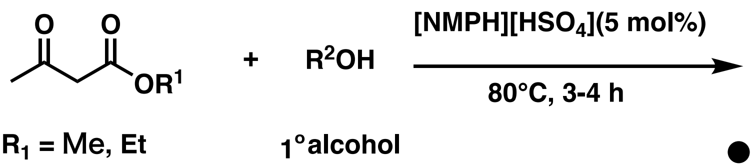
General Acid-Catalyzed Transesterification Mechanism

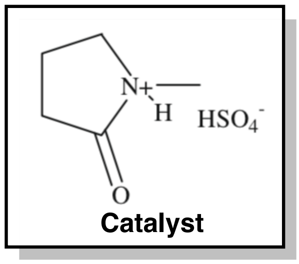
H_2SO_4 [11]

$$HC(OEt)_3 + 3ROH \xrightarrow[79^\circ C,\ 24\ h]{H_2SO_4} HC(OR)_3$$

R	n-Pr	n-butyl	isobutyl	s-butyl	n-amyl	isoamyl
Yield (%)	95.3	86.3	86.0	87.5	74.0	88.2

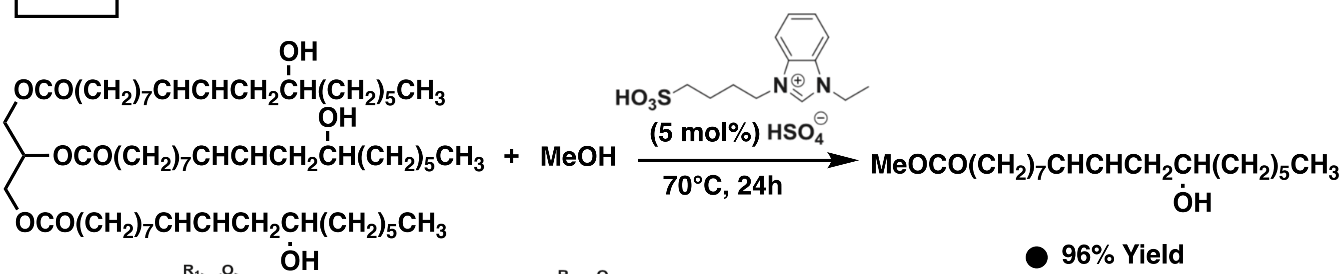
[NMPH][HSO_4] [12]





- 19 examples
- 70~91% Yield
- resue for four times

BBAIL [13]



- 96% Yield
- resue for three times

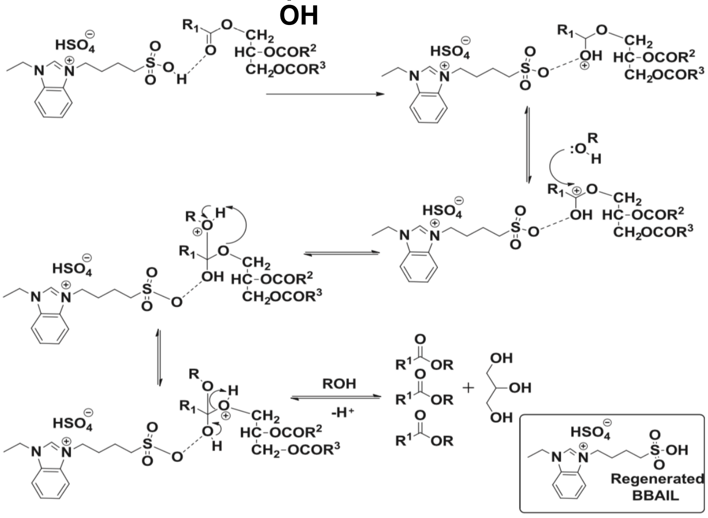


Table 4
Comparison of transesterification of nonedible oil using BBAIL with various reported catalysts.

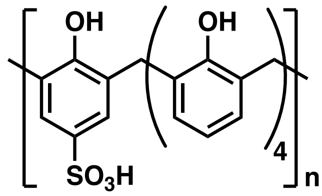
Entry	Catalyst for the castor oil transesterification	Catalyst loading	Yield (%)	Ref.
1	Bio-FeNPs catalyst	1 wt%	85.2	[39]
2	Si-Mica-Ph-SO_3H	5 wt%	90	[40]
3	Cesium doped heteropolyacid catalyst	2.94 w/w	90.5	[41]
4	Sulfonic acid functionalized benzimidazolium-based supported ionic liquid catalyst	3 wt%	94.9	[20]
5	Ni doped ZnO nanocatalyst	11% w/w	96.5	[42]
6	BBAIL	2.038 wt%	96	Present work

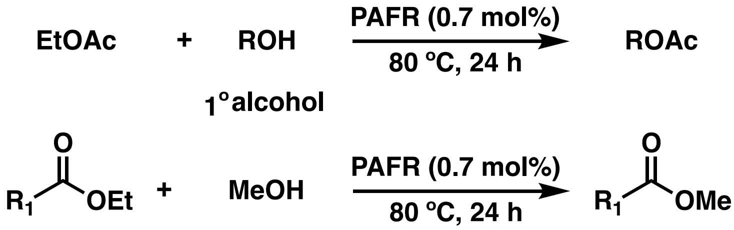
3. Catalysts

3-3. Brønsted Acid

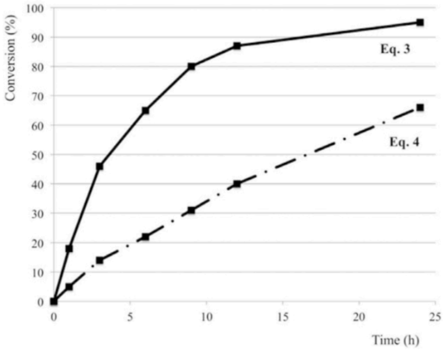
3-3-2. Heterogeneous Catalysts

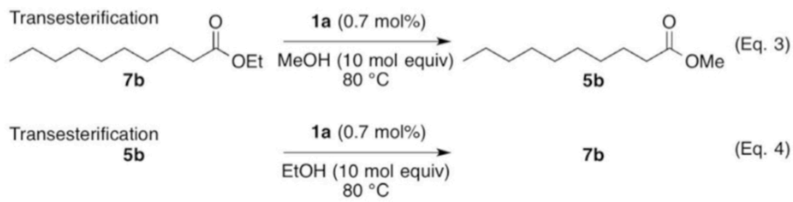
PAFR [14)]


PAFR

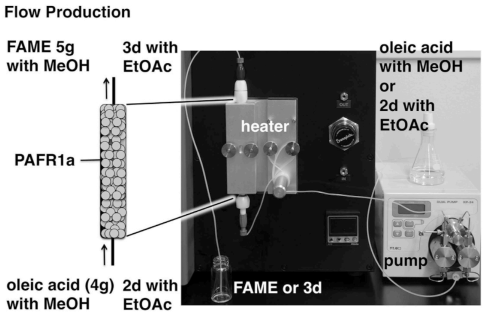


- 17 examples
- 78~97% Yield
- application of batch and continuous-flow production

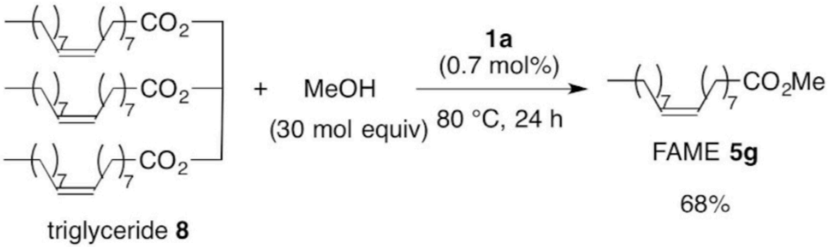




⟹ **The equilibrium is not an important factor when using PAFR , and thus the high catalytic activity is important for full conversion.**

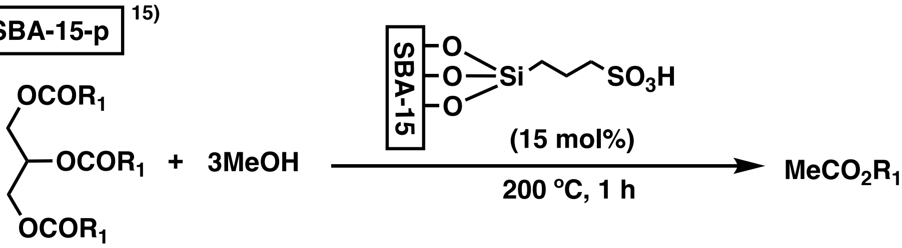


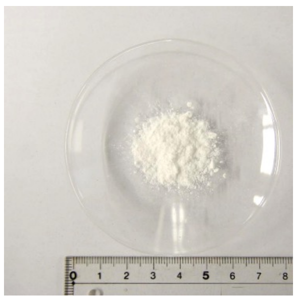
Yield of **5g** (continuous flow time): 92% (24 h), 92% (72 h), 92% (96 h)
Yield of **3d** (continuous flow time): 94% (12 h), 94% (57 h), 94% (111 h), 94% (152 h)



⟸ **Continuous-Flow synthesisi of biodiesel fuel (FAME).**

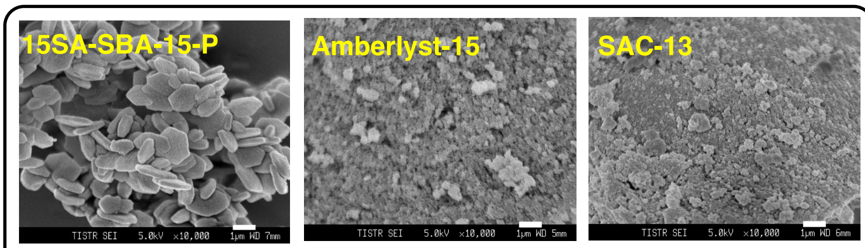
15SA-SBA-15-p [15)]



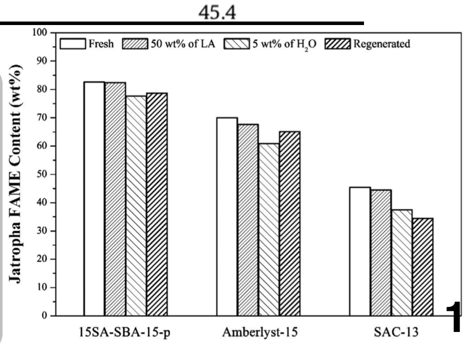


Physicochemical properties of 15SA-SBA-15-p, SAC-13 and Amberlyst-15 catalysts and their catalytic activities in Jatropha BDF synthesis.[a]

Catalysts	S_{BET} ($m^2\ g^{-1}$)	V_{Total} ($cm^3\ g^{-1}$)	Φ_p (nm)	Acid capacity[b] (mmol H^+ g-catal^{-1})	FAME[c] (wt%)
None	–	–	–	–	15.1
15SA-SBA-15-p	589	0.96	7.5	2.44	82.6
Amberlyst-15	21	0.32	72	4.50	70.0
SAC-13	215	0.57	57	1.35	45.4



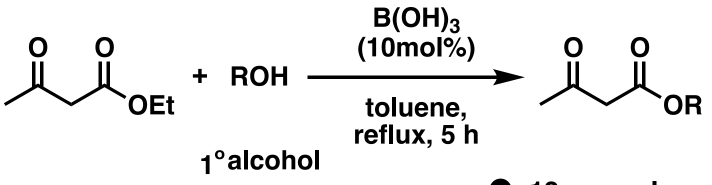
HRSEM Photos



3. Catalysts

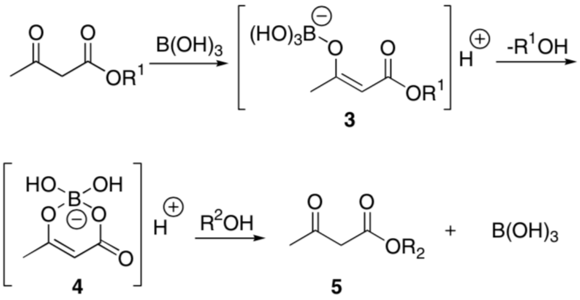
3-4. Lewis Acid

$B(OH)_3$ 16)



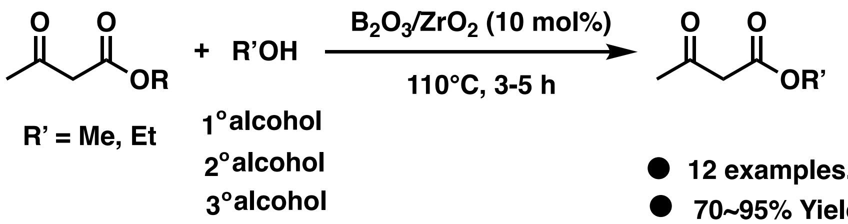
- 12 examples
- 89~99% Yield

Proposed Mechanism



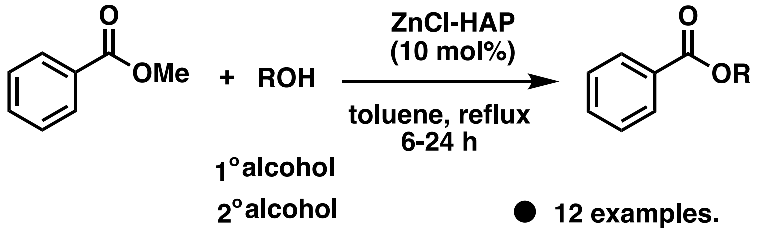
Scheme 3. Possible mechanism for the boric acid catalyzed transesterification.

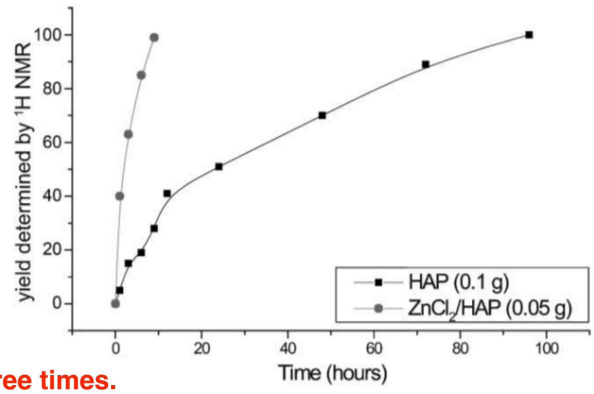
B_2O_3/ZrO_2 17)



- 12 examples.
- 70~95% Yield
- reuse for three times.

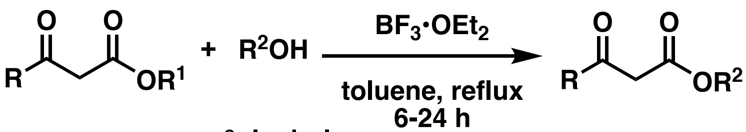
ZnCl-HAP 18)





- 12 examples.
- 89~99% Yield
- reuse at least three times.

$BF_3 \cdot OEt_2$ 19)



R = Me, Ph, allyl…
R^1 = Me, Et

1°alcohol
2°alcohol

- 12 examples.
- 89~99% Yield

Table 1. Transesterification for methyl acetoacetate with *n*-butanol under different conditions[a]

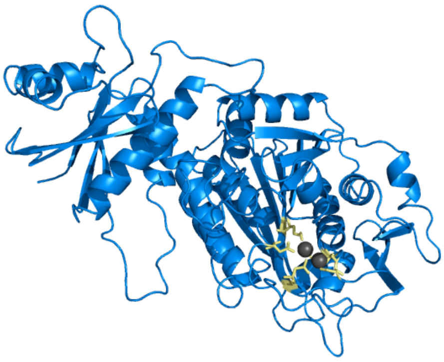
Entry	Catalysts	Time (h)	Yield (%)[b]
1	—	15	0
2	$Fe_2(SO_4)_3$ (10 mol%)	4.5	80
3	$ZnCl_2$ (10 mol%)	6.0	67
4	$NiCl_2$ (10 mol%)	5.0	43
5	CuCl (10 mol%)	4.0	57
6	$CuCl_2$ (15 mol%)	7.0	54
7	$Bi(NO_3)_5$ (10 mol%)	5.0	47
8	$(NH_4)_2Ce(NO_3)_6$ (10 mol%)	5.5	55
9	$CeCl_3 \cdot 7H_2O$ (15 mol%)	4.0	63
10	$(NH_4)_6Mo_7O_{24}$ (20 mol%)	5.5	61
11	$CoCl_2$ (10 mol%)	5.5	51
12	$BF_3 \cdot OEt_2$ (10 mol%)	6.5	64
13	$BF_3 \cdot OEt_2$ (15 mol%)	5.0	92
14	$BF_3 \cdot OEt_2$ (20 mol%)	5.5	91

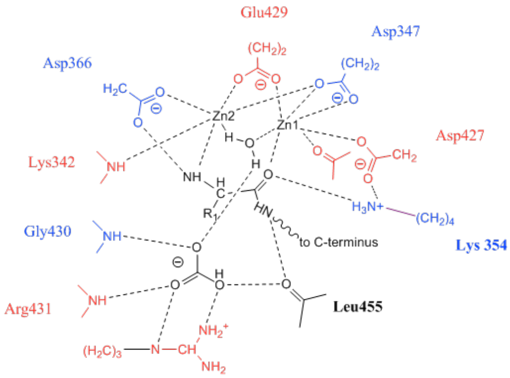
3. Catalysts

3-4. Lewis Acid

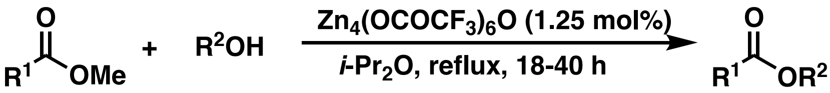
Zinc Cluster

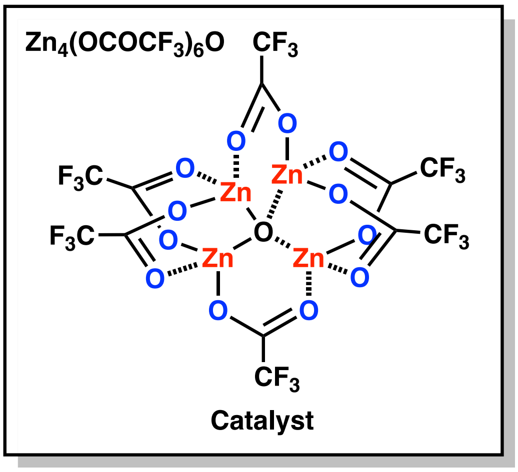
- Leucyl aminopeptidase
Enzymes catalyzed the hydrolysis of leucine residues at the N-terminus of peptides and proteins.





- Tetranuclear Zinc Cluster [20]





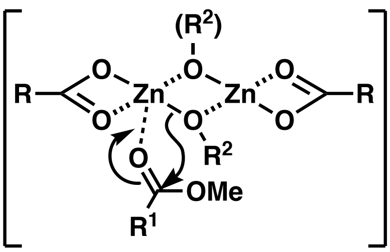
Advantage
- approximately equal amounts
- low amounts of catalyst

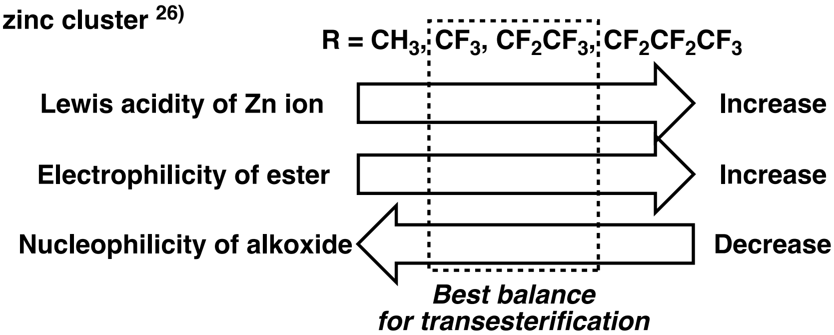
Disadvantage
- long reaction time (18-40 h)
- explosiveness solvent

Substrate scope
- 1°, 2°-alcohols
- *N*-protected α-amino esters[21)]
  β-keto esters[22)], acrylate[23)]
- acetylation[24)], methanolysis[25)]

Substrate limitation
- 3°-alcohols
- acidic alcohols
  (phenol pK_a 9.95, HFIP pK_a 9.3)

- Electronic effect of tetranuclear zinc cluster [26)]

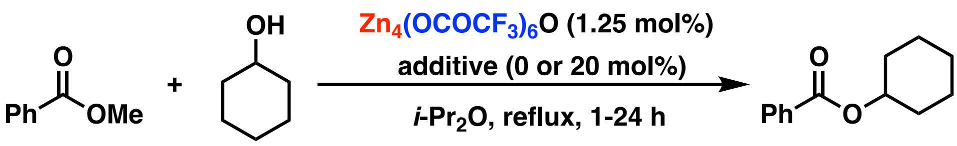


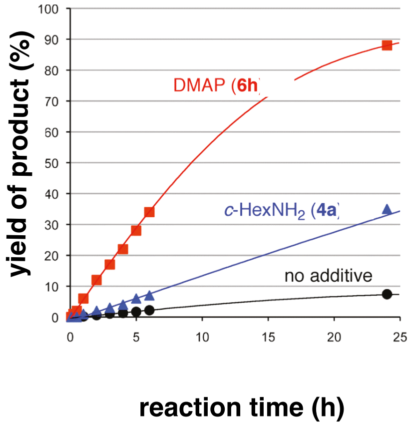


3. Catalysts

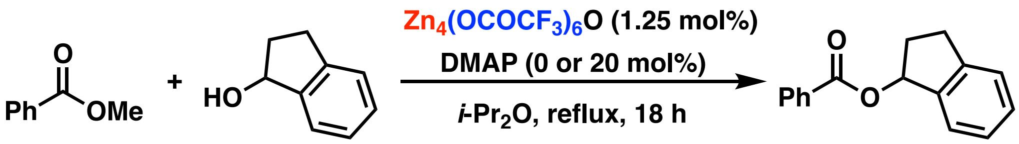
3-4. Lewis Acid

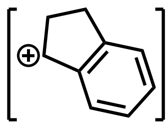
- **Additive effect of DMAP** [27]





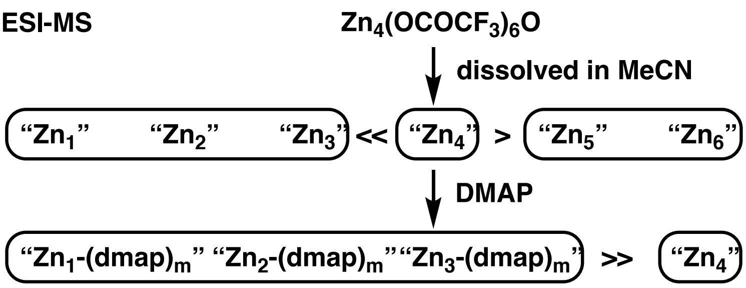
- **DMAP makes the neutral reaction conditons**





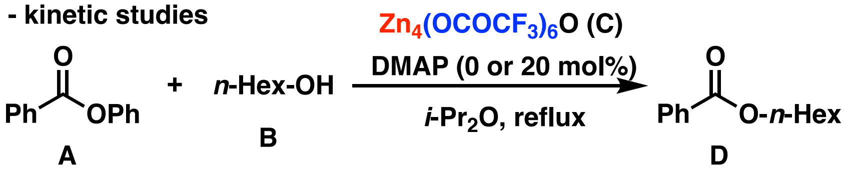
88% yield (with DMAP)
5% yield (without DMAP)

- **ESI-MS**



- **^{1}H NMR**
  Zn ions are coordinated by DMAP in preference to ester or alcohol

- **kinetic studies**



without DMAP

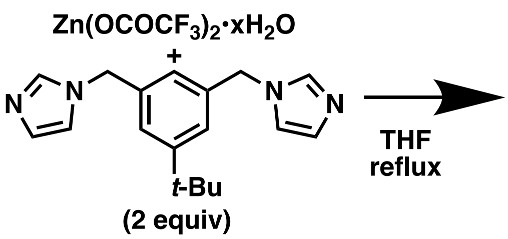
$$\frac{d[D]}{dt} = k[A]^{0.98}[B]^{0.66}[C]^{0.52}$$

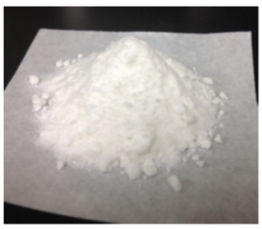
with DMAP

$[C]^{0.52}$ -> $[C]^{0.41}$

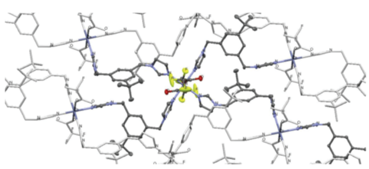
-the exact structure of the active species remains unclear… but, "Zn_2-(dmap)$_m$" or "Zn_3-(dmap)$_m$" is more active than other species. Reaction does not proceed via acyl pyridinium salt.

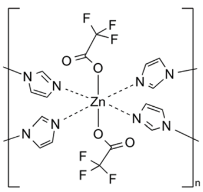
- **Bis(imidazole) Ligand** [28]





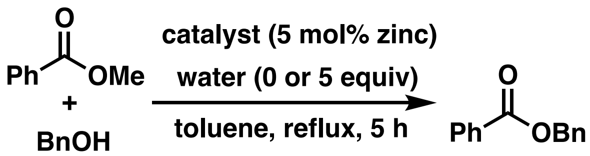
91%





X-ray crystallographic structure

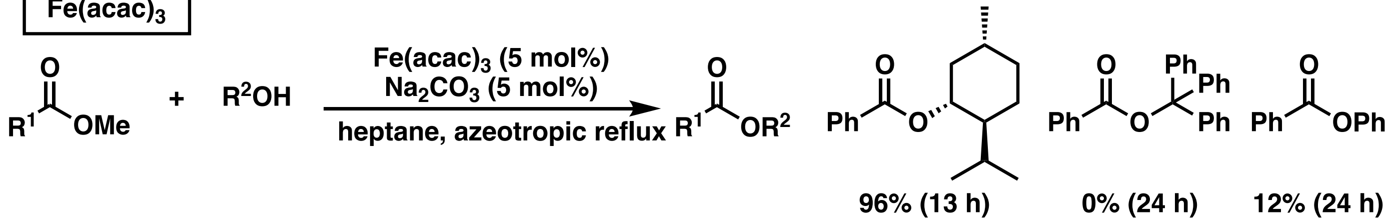
- heterogeneous catalyst - recovery and reuse - resistance to water



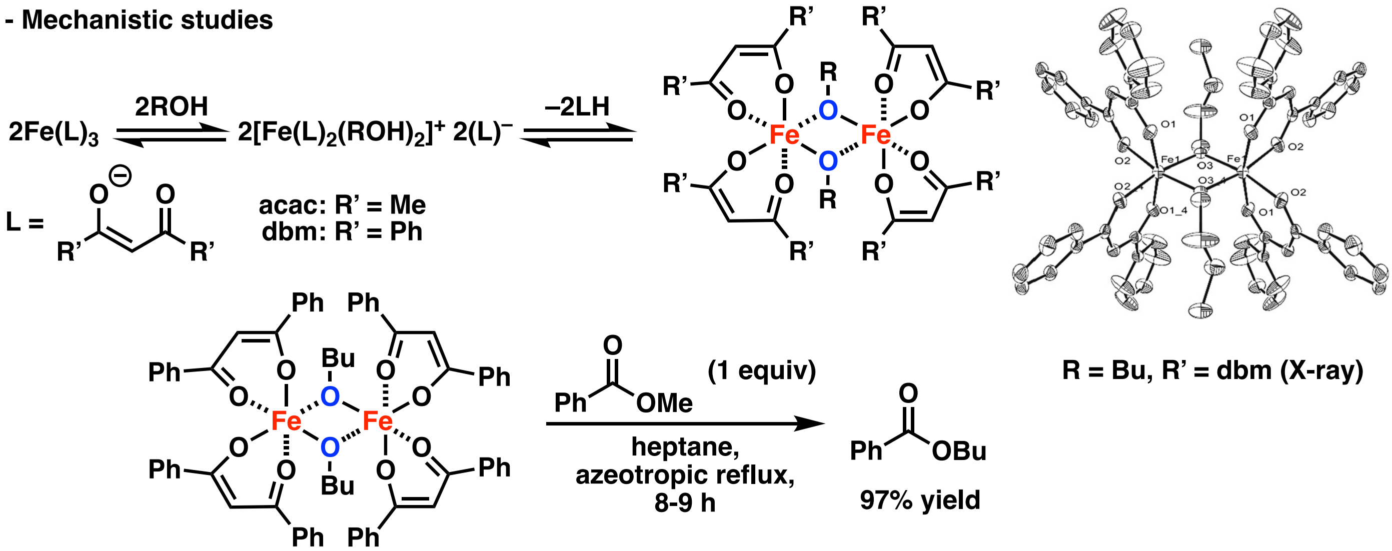
$Zn_4(OCOCF_3)_6O$	71%
$Zn_4(OCOCF_3)_6O$ + water	31%
Zn complex	100%
Zn complex + water	80% (100%, 21 h)

3-4. Lewis Acid

$Fe(acac)_3$ [29)]

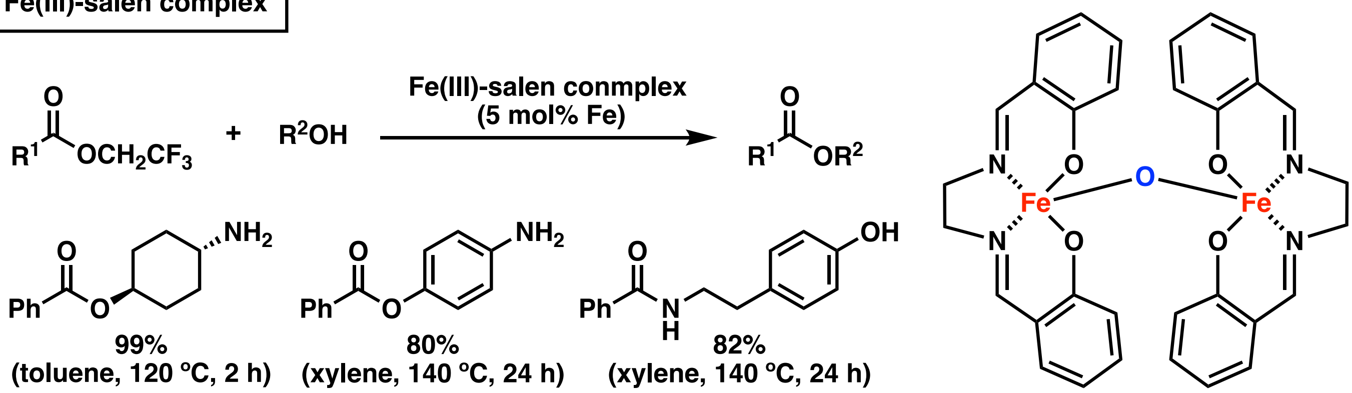


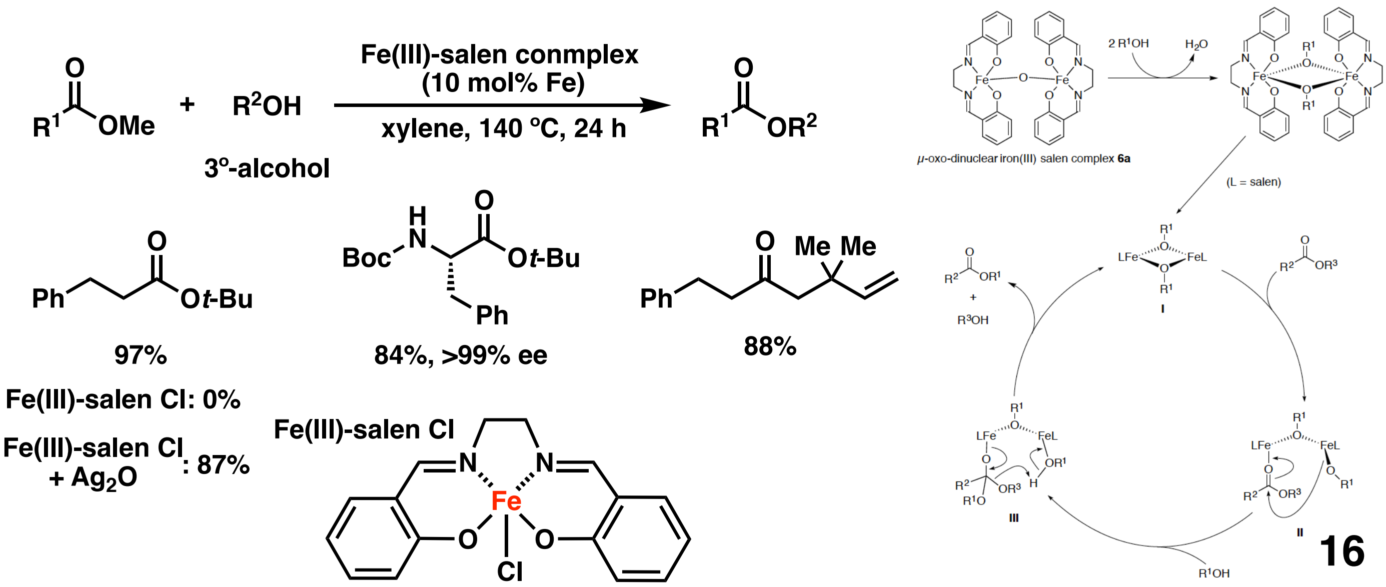
- Mechanistic studies



R = Bu, R' = dbm (X-ray)

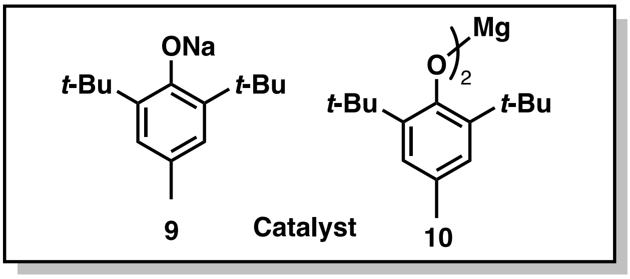
Fe(III)-salen complex [30)]





3-4. Lewis Acid

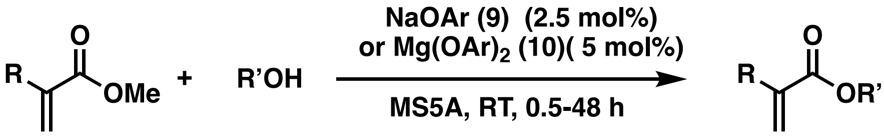
Mg(OAr)$_2$&NaOAr [31]



9 Catalyst 10

Ishihara, K.

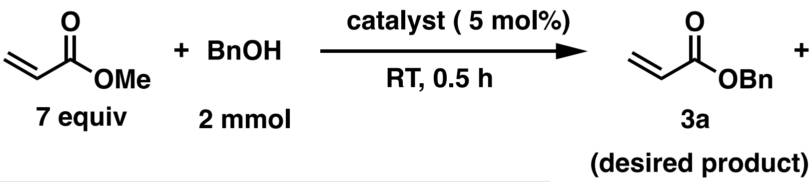
Hatano, M.

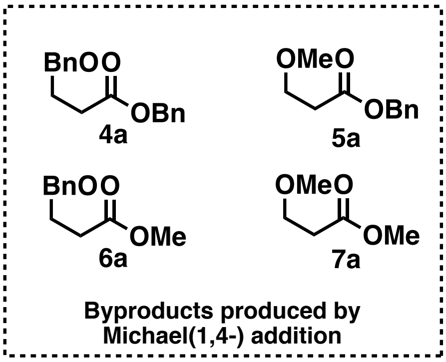


R = H, Me

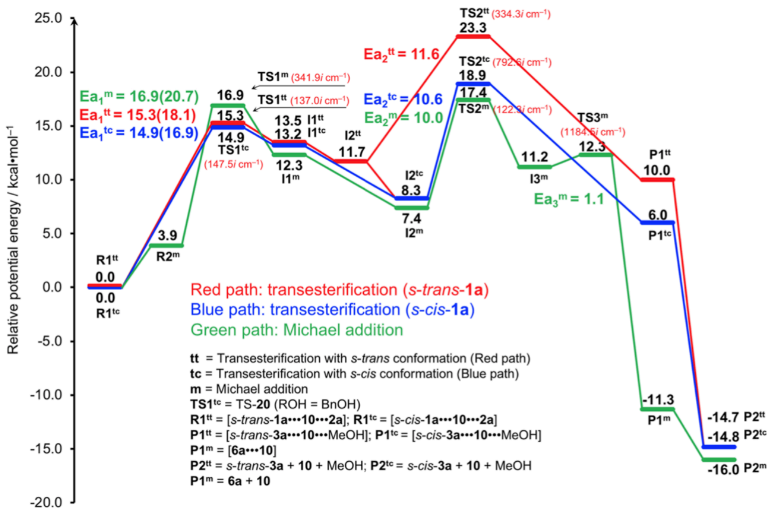
- 52 examples
- 40~99% Yield

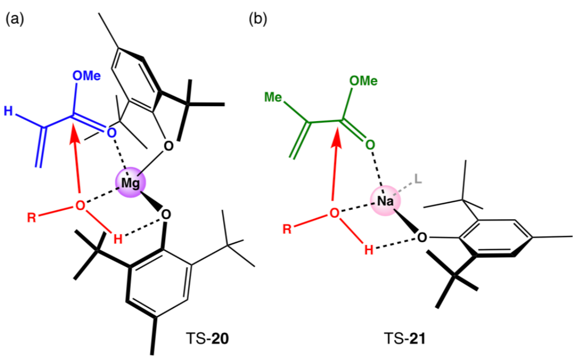
Screening of the catalysts





entry	catalyst	yield (%)[b]				
		3a	4a	5a	6a	7a
1	LiOAr	50	8	8	25	13
2	NaOAr (**9**)	49	8	0	38	55
3	KOAr	40	8	2	47	45
4	Mg(OAr)$_2$ (**10**)	**40**	**0**	**0**	**0**	**1**
5[c]	**10**	**70**	**0**	**0**	**1**	**4**
6	Ca(OAr)$_2$	6	0	0	1	1
7	NaOMe	43	8	1	41	40
8	KO*t*-Bu	20	10	17	30	30
9	Mg(O*t*-Bu)$_2$	60	1	0	8	15
10[c]	Ca(O*i*-Pr)$_2$	40	1	0	8	15
11[c]	Ti(O*n*-Bu)$_4$	0	0	0	0	0
12[c]	Fe(OEt)$_3$	0	0	0	0	0
13[c]	Zn(OMe)$_2$	0	0	0	0	0
14	La(O*i*-Pr)$_3$	62	5	10	18	32
15	$[Me_4N]^+[OCO_2Me]^-$ (**8**)	2	0	0	1	0
16	$[Me_4N]^+[OAr]^-$	34	10	0	54	42





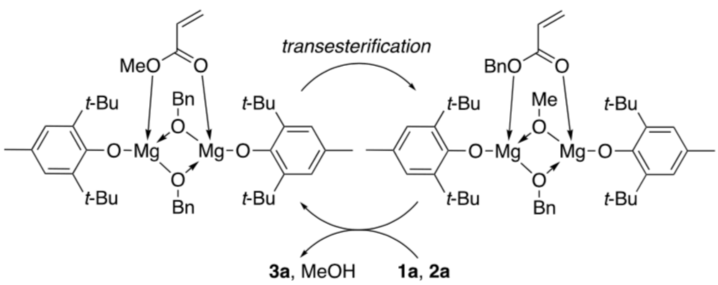


Figure 6. Possible reaction mechanism including dimeric Mg(II) complexes.

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