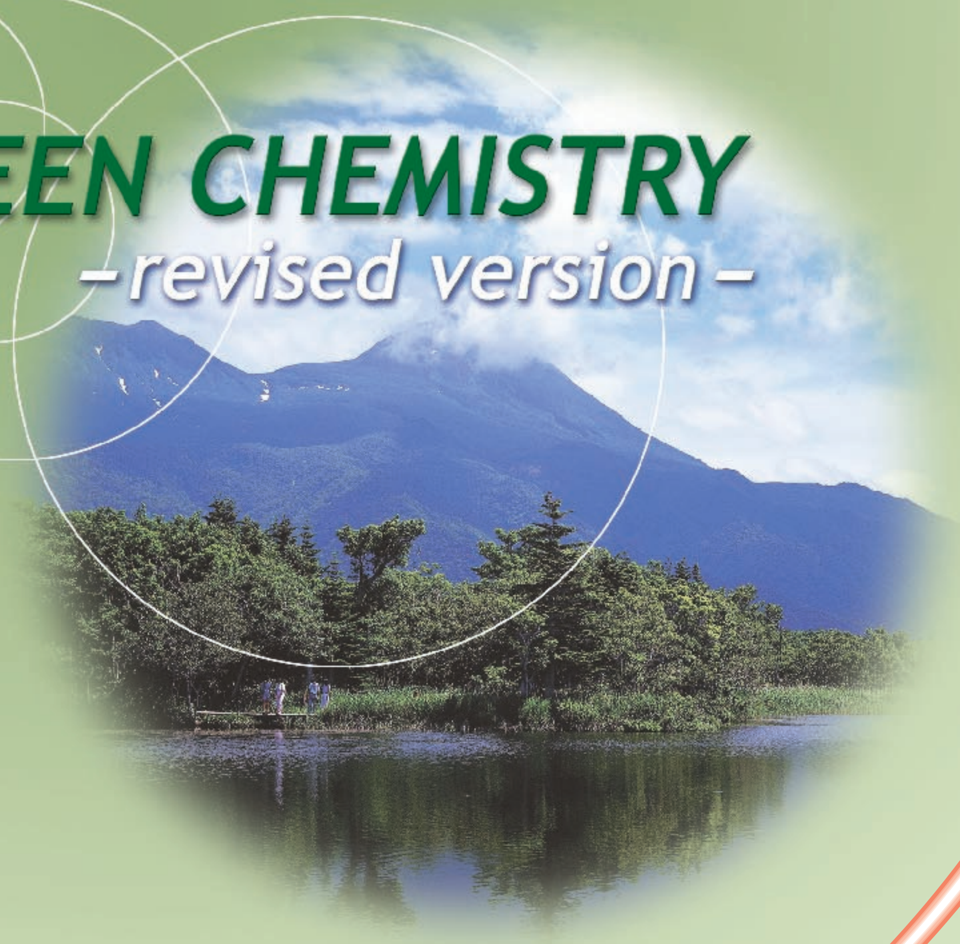
Wako Product Update

No.9

GREEN CHEMISTRY
– revised version –



Please visit the Wako Online Catalog
http://search.wako-chem.com

Wako

Organic Chemistry
- Catalysts
- Ligands for Catalytic Asymmetric Synthesis
- Others

Analytical Chemistry
- Paper Fiber-made TLC sheet

2004

Index

☞ See page #28

A. Metal Catalysts

	description		Page
Co	Metallocene	Cobaltocene	16
Co	Polymer-supported Catalyst	Dichlorobis(triphenylphosphine)cobalt(II), Supported PS Resin	4
Cr	Metallocene	Bis(pentamethylcyclopentadienyl)-chromium (II)	16
Hf	Metallocene	Bis(butylcyclopentadienyl)hafnium (IV) Dichloride	16
Hf	Metallocene	Bis(cyclopentadienyl)hafnium (IV) Dichloride	16
Ni	Polymer-supported Catalyst	Dibromobis(triphenylphosphine)nickel (II), Supported PS Resin	4
Os	Polymer-supported Catalyst	Dipotassium Dioxotetrahydroxoosmate (VIII)-Triethylamine PE fibres	9
Os	Polymer-supported Catalyst	FibreCat®3003, 3004	9
Os	Polymer-supported Catalyst	Osmium (VIII) Oxide, Microencapsulated	3
Os	Polymer-supported Catalyst	Osmium (VIII) Oxide, Microencapsulated PEM	3
Os	Polymer-supported Catalyst	Osmium (VIII) Oxide-Pyridine PE fibres	9
Pd	Polymer-supported Catalyst	Acetonitrile Dichloropalladium (II)-Triphenylphosphine PE fibres	9
Pd	Polymer-supported Catalyst	Di-μ-chlorobis [(η-allyl) palladium (II)], Supported PEG-PS Resin	5
Pd	Polymer-supported Catalyst	FibreCat® 1001, 1007, 1026	9
Pd	Polymer-supported Catalyst	Palladium (II) Acetate-Dicyclohexylphenylphosphine PE fibres	9
Pd	Polymer-supported Catalyst	Palladium (II) Acetate-Triphenylphosphine PE fibres	9
Pd	Polymer-supported Catalyst	Palladium (II)-Hydrotalcite	5
Pd	Polymer-supported Catalyst	Palladium (II)-Hydrotalcite (modified)	5
Pd	Stable Pd/C Catalyst	Palladium-Activated Carbon Ethylenediamine Complex (Pd: 5%)	6
Pd	Catalyst acted in Water	Palladium-Nanocage	11
Pd	Polymer-supported Catalyst	PEG-PS resin-supported phosphine-Palladium complex	5
Pd	Polymer-supported Catalyst	PEP-Pd	5
Pd	Polymer-supported Catalyst	Tetrakis(triphenylphosphine)palladium(0), Supported PS Resin	4
Pd	Polymer-supported Catalyst	Triphenylphosphine Pd (0), Microencapsulated	4
Pt	Polymer-supported Catalyst	cis-Bis(acetonitrile)dichloroplatinum (II)-Triphenylphosphine PE fibres	9
Pt	Polymer-supported Catalyst	Dihydrogen Hexachloroplatinate (IV) n-Hydrate-Pyridine PE fibres	9
Pt	Polymer-supported Catalyst	FibreCat® 4001, 4003	9
Rh	Polymer-supported Catalyst	Bis(η-norbornadiene)rhodium (I) Tetrafluoroborate-Triphenylphosphine PE fibres	9
Rh	Polymer-supported Catalyst	Di-μ-chlorobis (η-norbonadiene)dirhodium (I)-Triphenylphosphine PE fibres	9
Rh	Polymer-supported Catalyst	FibreCat® 2003, 2006	9
Ru	Polymer-supported Catalyst	FibreCat® 3002	9
Ru	Catalyst	met-DIRUX	8
Ru	Catalyst	Methanethiolate-Bridged Diruthenium Complex	8
Ru	Polymer-supported Catalyst	RuHAP	7
Ru	Polymer-supported Catalyst	Ruthenium(III)-Hydroxyapatite	7
Ru	Metallocene	Ruthenocene	16
Ru	Polymer-supported Catalyst	Sodium Ruthenate (VII)-Triethylamine PE fibres	9
Sc	Catalyst acted in Water	Scandium (III) Trifluoromethanesulfonate	11
Sc	Catalyst acted in Water	Scandium Tris(dodecyl sulfate)Trihydrate	12

Catalysts

Catalysts

Sc	Polymer-supported Catalyst	Scandium Trifluoromethanesulfonate, Microencapsulated	4
	Catalyst acted in Water	STDS	12
Ti	Metallocene	Bis(butylcyclopentadienyl)titanium (IV) Dichloride	16
	Metallocene	Bis(cyclopentadienyl) titanium Dichloride	
	Metallocene	Bis(methylcyclopentadienyl)-titanium (IV) Dichloride	
	Metallocene	Bis(t-butylcyclopentadienyl)-titanium (IV) Dichloride	
	Metallocene	Ethylenebis(1-indenyl)titanium (IV) Dichloride	
W	Metallocene	Bis(cyclopentadienyl)tungsten (IV) Dichloride	
	Solid Super-Acid Catalyst	Zirconia, Tungstate	19
	Solid Super-Acid Catalyst	Zirconia, Tungstate, Pellet	
Zr	Metallocene	Bis(cyclopentadienyl)dimethyl-zirconium (IV)	16
	Metallocene	Bis(cyclopentadienyl)zirconium (IV) Chloride Hydride	
	Metallocene	Bis(dodecylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(ethylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(indenyl)zirconium (IV) Dichloride	
	Metallocene	Bis(methylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(n-butylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(octylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(propylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(t-butylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Bis(trimethylsilylcyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	(Cyclopentadienyl)(indenyl)-zirconium (IV) Dichloride	
	Metallocene	Dimethylsilylenebis(cyclopentadienyl)-zirconium (IV) Dichloride	
	Metallocene	Diphenylmethylidene(cyclopenta-dienyl)(9-fluorenyl)zirconium (IV) Dichloride	
	Metallocene	meso-Dimethylsilylenebis(2-methyl-1-indenyl)zirconium (IV) Dichloride	17
	Metallocene	rac-Dimethylsilylenebis(1-indenyl)-zirconium (IV) Dichloride	
	Metallocene	rac-Dimethylsilylenebis(2-methyl-1-indenyl)zirconium (IV) Dichloride	
	Metallocene	rac-Dimethylsilylenebis(4,5,6,7-tetrahydro-1-indenyl)zirconium (IV) Dichloride	
	Metallocene	meso-Ethylenebis(1-indenyl)-zirconium (IV) Dichloride	16
	Metallocene	rac-1,2-Ethylenebis(2-methyl-1-indenyl)zirconium (IV) Dichloride	17
	Metallocene	rac-Ethylenebis(1-indenyl)-dimethylzirconium(IV)	
	Metallocene	rac-Ethylenebis(4,5,6,7-tetrahydro-1-indenyl)zirconium (IV) Dichloride	
	Metallocene	Isopropylidene(3-methylcyclopenta-dienyl)(9-fluorenyl)zirconium (IV) Dichloride	16
	Metallocene	Isopropylidene(cyclopentadienyl)-(9-fluorenyl)zirconium (IV) Dichloride	
	Metallocene	Isopropylidenebis(cyclopentadienyl)-zirconium (IV) Dichloride	
	Solid Super-Acid Catalyst	Zirconia, Sulfated	19
	Solid Super-Acid Catalyst	Zirconia, Sulfated, Pellet	
	Solid Super-Acid Catalyst	Zirconia, Tungstate	
	Solid Super-Acid Catalyst	Zirconia, Tungstate, Pellet	
	Metallocene	Zirconocene Dichloride	16

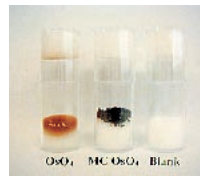
B. Polymer-supported catalysts

a. Microencapsulated Catalysts

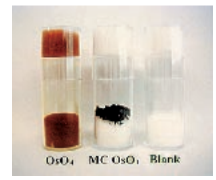
[Features]

1. **Readily recoverable and reusable by filtration**
2. **High catalytic activity**
3. **Utilizing patented technology that reduces release of catalyst from resin**
4. **Environmentally friendly**

Low Toxicity due to Low Volatility



[Before]



[After 3 days]

Microencapsulated OsO_4, prepared from polystyrene based on the microencapsulated technique

Osmium (VIII) oxide, Microencapsulated is a useful, safe, non-volatile, recoverable and reusable catalyst for the asymmetric dihydroxylation of olefins on an industrial scale.

Osmium (VIII) oxide, Microencapsulated (MC OsO_4)

Os

Cat. #153-02081 **1 g**

RT, Solid (black chipped mass)

MW : 254.23 (OsO_4)

CAS:20816-12-0

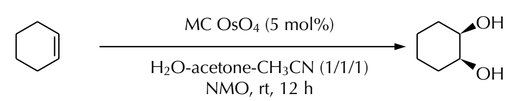
OsO_4 content : approx. 10 %

IATA : Not restricted

[Application]

Best applied when N-methylmorpholine-N-oxide (NMO) is used as a cooxidant.

[Reaction] **Recovery and reuse of MC OsO_4**



Run	1	2	3	4	5
Yield of Product (%)	84	84	83	84	83
Recovery of Catalyst (%)	quant.	quant.	quant.	quant.	quant.

Osmium (VIII) oxide, PEM Microencapsulated (PEM-MC OsO_4)

Os

Cat. #158-02411 **1 g**

RT, Solid (black chipped mass)

MW : 254.23 (OsO_4)

CAS:20816-12-0

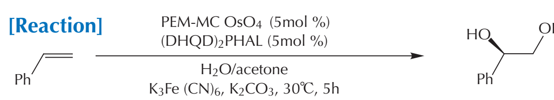
OsO_4 content : approx. 10 % (Fluorescent X-rays analysis)

IATA : Not restricted

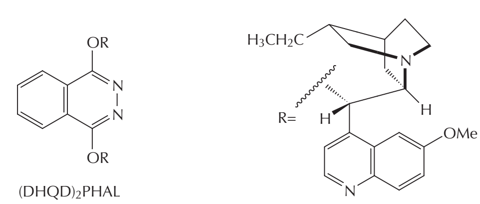
[Application]

PEM-based microencapsulated osmium (VIII) oxide is hydrophilic. Best applied when potassium ferricyanide ($K_3[Fe(CN)_6]$) is used as a cooxidant. When an asymmetric ligand is used, PEM- MC OsO_4 can be applied for asymmetric dihydroxylation reaction without the slow addition of olefins.

[Reaction]



Run	1	2	3
Yield of Product (%)	85	66	84
ee (%)	78	78	78
Recovery of Catalyst (%)	quant.	quant.	quant.



(DHQD)$_2$PHAL

[References]

1) S. Kobayashi, M. Endo and S. Nagayama: *J. Org. Chem.*, **63**, 6094 (1998).
2) S. Kobayashi, T. Ishida and R. Akiyama: *Org. Lett.*, **3**, 2649 (2001).

Wako will perform contract services to supply microencapsulated osmium (VIII) oxide on an industrial scale and to synthesize diols and asymmetric diols.

Catalysts

(a. Microencapsulated Catalysts)

Sc Scandium Trifluoromethanesulfonate, Microencapsulated

Cat. #196-12041 1 g
RT, Solid

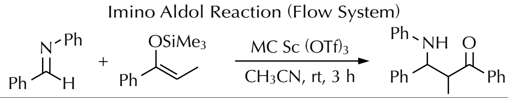
MW : 492.15 ($C_3F_9O_9S_3Sc$)
CAS : 144026-79-9
Appearance : White mass
$Sc(OTf)_3$ content : approx. 10 %

[Features]
1. **Readily recoverable and reusable by filtration**
2. **Usable in both batch and flow system**
3. **Hardly any elution of Sc(OTf)3**

Scandium Trifluoromethanesulfonate (Sc(OTf)3), a Lewis acid enabling to use water as a solvent for a wide range of organic reactions. Wako offers the polystyrene resin-supported catalyst based on the microencapsulation technique.

[Reaction 1]

Imino Aldol Reaction (Flow System)

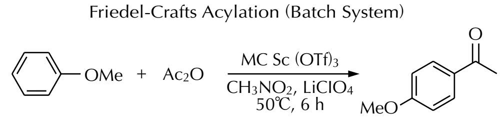


Use[a]	1	2	3	4	5	6	7
Yield/%	90	90	88	89	89	88	90

[a]Recovered catalyst was used successively (Use 2,3,4....)

[Reaction 3]

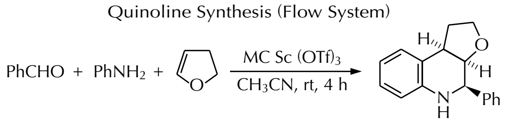
Friedel-Crafts Acylation (Batch System)



1st use, 76% yield ; 2nd use, 76% yield ; 3rd use, 81% yield

[Reaction 2]

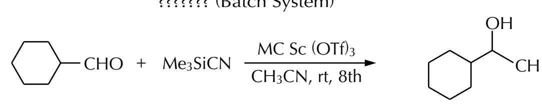
Quinoline Synthesis (Flow System)



1st use, 68% yield ; 2nd use, 69% yield ; 3rd use, 69% yield

[Reaction 4]

??????? (Batch System)



1st use, 79% yield ; 2nd use, 78% yield ; 3rd use, 74% yield

[Other Reactions]

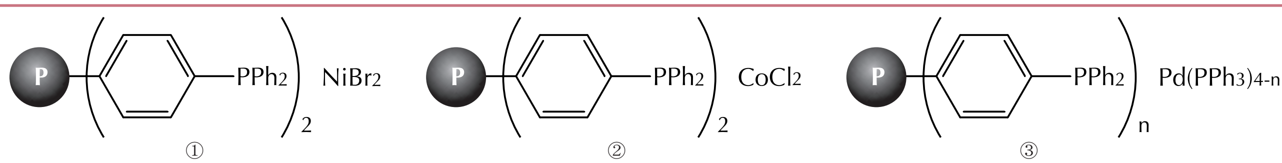
1. By using imines : AzaDiels-Alder reaction, Allylation reaction
2. Three-component condensation reaction : Mannich-type reaction, Stretcher reaction
3. By using carbonyl compounds : Aldol reaction, Michael reaction, Cyanation reaction, Diels-Alder reaction

b. Triphenylphosphinated polystyrene complex

Catalyst made by fixing metals (Pd, Ni and Co) with the triphenylphosphine structure on polystyrene. This triphenylphosphine structure is bound not by a linker but in a form including the benzene ring of polystyrene. It is therefore relatively stable in various reactions.

[Features]
1. **Roughly homogeneous-sized particles with an average diameter of approximately 60μm, enabling efficient reaction**
2. **Readily recoverable and reusable by filtration**

[structural formula]



Product List

	Catalog No.	Product	Package Size	Content		Structure
Ni	042-28421	Dibromobis(triphenylphosphine)nickel (II), Supported PS Resin	500 mg	Ni : 0.87 mmol/g	2~10℃, Solid	①
Co	049-28431	Dichlorobis(triphenylphosphine)cobalt(II), Supported PS Resin	500 mg	Co : 0.95 mmol/g	2~10℃, Solid	②
Pd	205-15561	Tetrakis(triphenylphosphine)palladium(0), Supported PS Resin	500 mg	Pd : 0.07 mmol/g	2~10℃, Solid	③

c. Palladium Catalysts

Di-μ-chlorobis [(η-allyl) palladium (II)], Supported PEG-PS Resin

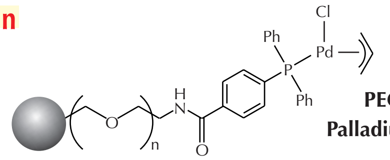
[PEG-PS resin-supported phosphine-Pd complex; PEP-Pd]

Cat. #043-27731 500 mg

2~10℃, Solid (yellow sticky mass)

Amphiphilic Resin-Supported Pd-Phosphine Catalyst

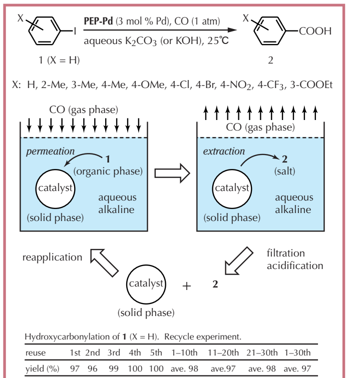
Pd



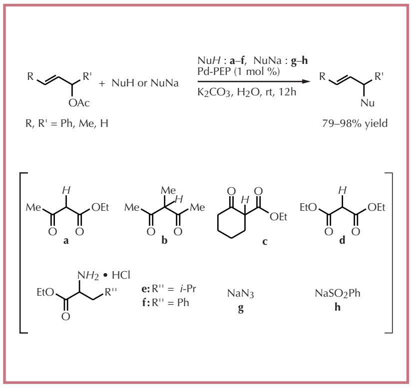
PEG-PS resin-supported Palladium-Phosphine Complex (Pd-PEP)

[Features]

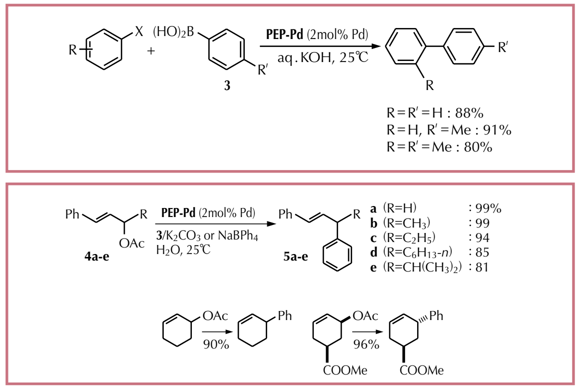
1. Readily recoverable and reusable by filtration for over 30 times
2. Amphiphillic resin-supported phosphine-palladium complex
3. Exhibits high catalytic activities in Tuji-Trost reaction, Suzuki-Miyaura cross-coupling reaction and Heck reaction in water by using PEP-Pd



Hydroxycarbonylation of aryl halides in water catalyzed by PEP-Pd [2)]



High catalytic activity of PEP-Pd in the allylic substitution in water [1)]



Cross-coupling of aryl halides and ally acetates with arylboron reagents in water catalyzed by PEP-Pd [3)]

[References]

1) Allylic substitution: (a)Uozumi, Y., Danjo, H., and Hayashi, T., *Tetrahedron Lett.*, **38**, 3557-3560 (1997), (b)Uozumi, Y., *et al., Tetrahedron Lett.*, **39**, 8303-8306 (1998), (c)Danjo, H., *et al., Tetrahedron*, **55**, 14341-14352 (1999).
2) Hydroxycarbonylation: Uozumi, Y., Watanabe, T., *J. Org. Chem.*, **64**, 6921-6923 (1999).
3) Cross-coupling: Uozumi, Y., *et al., J. Org. Chem.*, **64**, 3384-3388 (1999).

Palladium (II)-Hydrotalcite (Pd : 1.5 %)

Cat. #161-20543 5 g

RT, Solid (pale yellow powder)

Palladium (II)-Hydrotalcite (m*) (Pd : 0.8%)

Cat. #168-20553 10 g

RT, Solid (pale yellow powder)

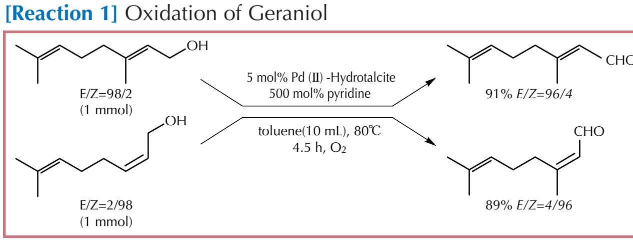
＊m : modified

[Features]

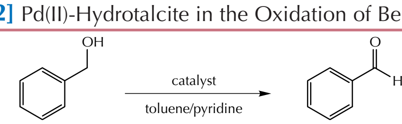
1. Pd(II)-hydrotalcite is a heterogeneous palladium catalyst that homogeneous palladium complex is immobilized on hydrotalcite ($Mg_6Al_2(OH)_{16}CO_3 \cdot H_2O$) which is a naturally produced basic clay mineral.
2. The noble immobilized Pd catalyst is found to be effective for the oxidation of a wide range of alcohols, using atmospheric oxygen or air.
3. In this catalytic system, various alcohols are readily converted to the corresponding aldehydes or ketones with high chemoselectivity or regioselectivity.

[Reaction 1] Oxidation of Geraniol

Pd



[Reaction 2] Pd(II)-Hydrotalcite in the Oxidation of Benzyl alcohol



entry	catalyst	gas	time (h)	number of use	GLC yield (%)
1a	Pd(II)-hydrotalcite	O_2	2	first second third	98 89 77
2b	Pd(II)-hydrotalcite	air	3	first second	98 90
3c	Pd(II)-hydrotalcite(m)	O_2	5	first second third	96 97 90

a) reaction conditions; Pd(II)-hydrotalcite (900 mg, 0.15 mmol Pd), benzyl alcohol (3.0 mmol), pyridine (1.5 mmol), toluene (30 mL), 80℃, O_2

b) reaction conditions; Pd(II)-hydrotalcite (300 mg, 0.05 mmol Pd), benzyl alcohol (1.0 mmol), pyridine (0.2 mmol), toluene (10 mL), 65℃, air (1 atm)

c) reaction conditions; Pd(II)-hydrotalcite(m) (1800 mg, 0.15 mmol Pd), benzyl alcohol (3.0 mmol), pyridine (1.5 mmol), toluene (30 mL), 80℃, O_2

[References] 1) T. Nishimura, N. Kakiuchi and S. Uemura: *Chem. Commun.***2000**, 1245-1246 (2000)
2) N. Kakiuchi, T. Nishimura, M. Inoue and S. Uemura: *Bull. Chem. Soc. Jpn.*, **74**, 165 (2001).

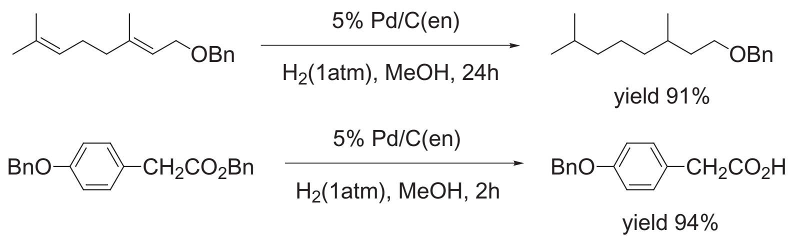
Catalysts

Palladium-Activated Carbon Ethylenediamine Complex (Pd 5%) [Pd/C (en)]

5% Pd/C (en) is a heterogeneous catalyst, whose structure is a 1:1 complex of the Pd/C's Pd and ethylenediamine. It enables a selective catalytic reduction of various functional groups under neutral conditions. It can be readily removed by filtration after the reaction.
5% Pd/C (en) is an excellent reduction catalyst that is not combustible, unlike the general Pd/C, and has good storage stability.
The development into industrial scale is anticipated.
In the catalytic reductions using 5% Pd/C (en), it is possible to easily reduce various functional groups, including olefin, azido, nitro, benzyl ester, aromatic hologen, etc., while inhibiting the reduction of protecting groups, such as benzyl ether[1], Cbz (benzyloxycarbonyl) group[1), 2)] which is an aliphatic amine, *O*-TBDMS (*t*-butyldimethylsilyl) group[3], epoxide[5] and benzyl alcohol[5].

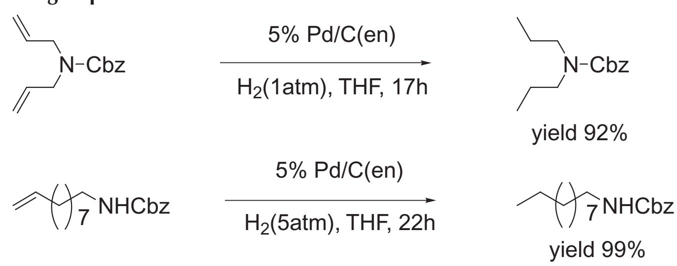
[Reaction 1]

Selective reduction in the presence of benzyl ether group[1)]



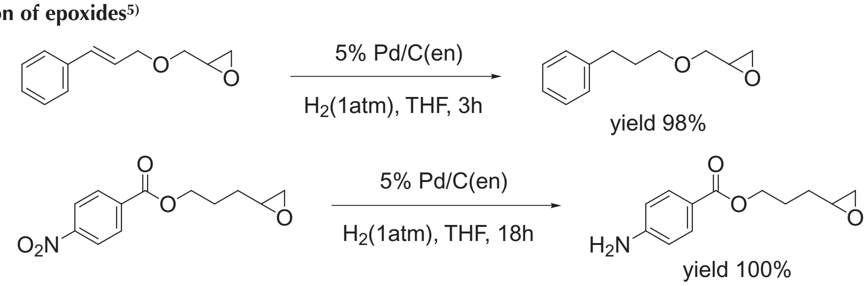
[Reaction 2]

Selective reduction in the presence of Cbz group[3),4)]



[Reaction 3]

Chemoselective hydrogenation of epoxides[5)]



Catalog No.	Description	Grade	Package Size	Storage
163-21441	Palladium-Activated Carbon Ethylenediamine Complex (Pd 5%)	for Organic Synthesis	1 g	Protect from light
169-21443			5 g	

[References]

1) H. Sajiki, K. Hattori, K. Hirota : *J. Org. Chem.*, **63**, 7990 (1998).
2) K. Hattori, H. Sajiki, K. Hirota : *Tetrahedron*, **56**, 8433 (2000).
3) K. Hattori, H. Sajiki, K. Hirota : *Tetrahedron Lett.*, **41**, 5711 (2000).
4) H. Sajiki, K. Hattori, K. Hirota : *Chem. Eur. J.*, **6**, 2200 (2000).
5) H. Sajiki, K. Hattori, K. Hirota : *J. Chem Soc., Perkin Trans.* **1**, 4043 (1998).

Pd-Nanocage …See page #11

d. HAP supported catalyst

Ruthenium(III)-Hydroxyapatite (RuHAP)

for Organic Synthesis

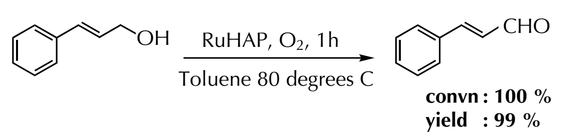
Cat. # 182-01851 1 g
188-01853 5 g
RT, Solid

RuHAP, developed by Prof. Kiyotomi Kaneda of Osaka Univ., is a novel, bound oxidation catalyst in the presence of molecular oxygen. Hydroxyapatite is a natural, inorganic crystallized compound. Using its surface as ligand, RuHAP is obtained by treating the surface with $RuCl_3$ aqueous solution. Because molecular oxygen (O_2) or air is used for reaction, it is unnecessary to use toxic heavy metals such as chromic acid or manganese dioxide, and clean oxidation can be carried out.

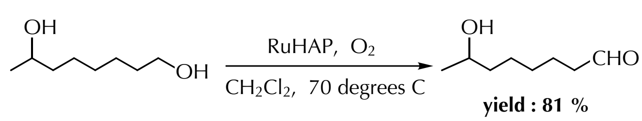
[Features]
1. High activity catalyst, 2. can be separated easily from products, 3. recyclable, 4. clean Aerobic oxidation without using toxic reagents

[Reactions]

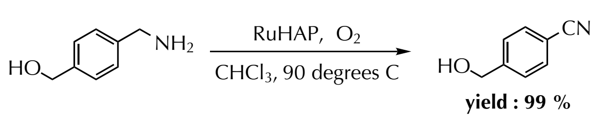
Oxidation of alcohols



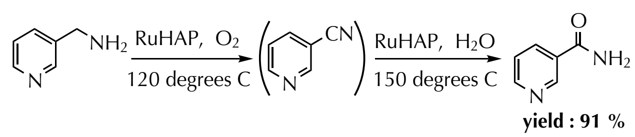
Intracellular competitive oxidation of primary and secondary alcohols



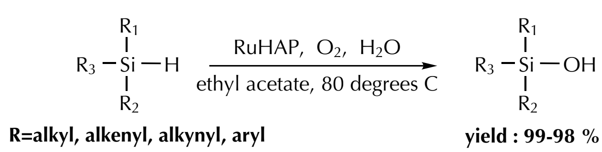
Intracellular competitive oxidation of amine and alcohol



Oxidation of amines



Oxidation of silanes



[Reference]

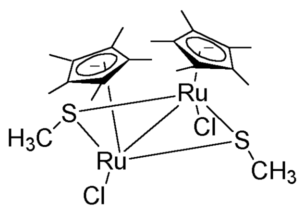
1) Yamaguchi, K., Mori, K., Mizugaki, T., Ebitani, K. and Kaneda, K.: *J. Am. Chem. Soc.*, **122**, 7144 (2000).
2) Mori, K., Yamaguchi, K., Mizugaki, T., Ebitani, K. and Kaneda, K.: *Chem. Commun.*, **461** (2001).
3) Mori, K., Tano, M., Mizugaki, T., Ebitani, K. and Kaneda, K.: *New J. Chem.*, **1536** (2002).

Catalysts

e. Methanethiolate-bridged diruthenium complex

met-DIRUX

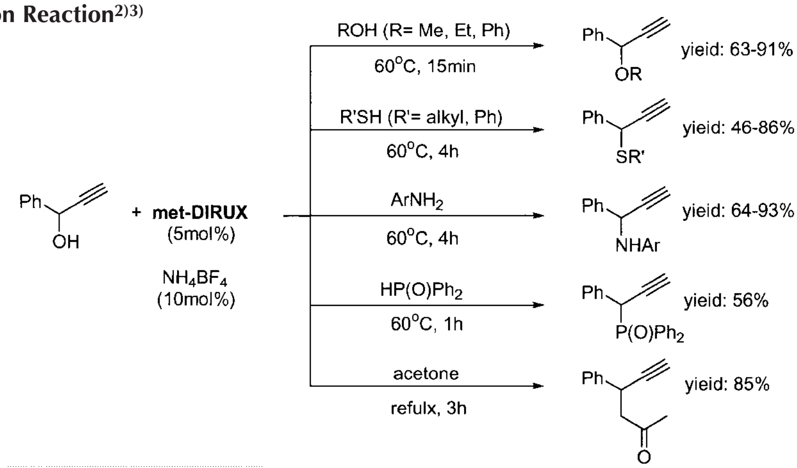
met-DIRUX is a chiral methanethiolate-bridged diruthenium catalyst in which pentamethylcyclopentadiene and methanethiol are coordinated to $RuCl_3$. It can catalytically react with various substrates via formation of ruthenium-allenylidene complexes with compounds such as propargyl alcohol. For example, it is applicable to cyclization of terminal diynes[1], propargylic substitution reaction of propargyl alcohol[2) 3)], cycloaddition with phenol derivatives[4], propargylation of aromatic compounds[5] and allenylidene-ene reaction with alkenes[6].
met-DIRUX is also applicable to asymmetric synthesis and is an air-stable catalyst.



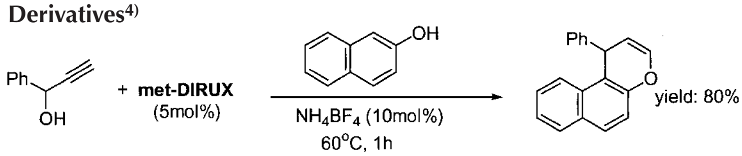
met-DIRUX :[Cp*RuCl(μ_2-SMe)$_2$RuCp*Cl]
(Methanethiolate-Bridged Diruthenium Complex)

[Reaction]

Propargylic Substitution Reaction[2)3)]



Cycloaddition with Phenol Derivatives[4]



Catalog No.	Description	Grade	Package Size	Storage
130-14581	met-DIRUX	For Organic Synthesis	200 mg	RT

[References]

1) Y. Nishibayashi, M. Yamanashi, I. Wakiji, M. Hidai : *Angew. Chem. Int. Ed.*, **39**, 2909 (2000).
2) Y. Nishibayashi, M. Yamanashi, I. Wakiji, M. Hidai : *J. Am. Chem. Soc.*, **122**, 11019 (2000).
3) Y. Nishibayashi, I. Wakiji, Y. Ishii, S. Uemura, M. Hidai : *J. Am. Chem. Soc.*, **123**, 3393 (2001).
4) Y. Nishibayashi, Y. Inada, M. Hidai, S. Uemura : *J. Am. Chem. Soc.*, **124**, 7900 (2002).
5) Y. Nishibayashi, M. Yoshikawa, Y. Inada, M. Hidai, S. Uemura : *J. Am. Chem. Soc.*, **124**, 11846 (2002).
6) Y. Nishibayashi, Y. Inada, M. Hidai, S. Uemura : *J. Am. Chem. Soc.*, **125**, 6060 (2003).

Catalysts

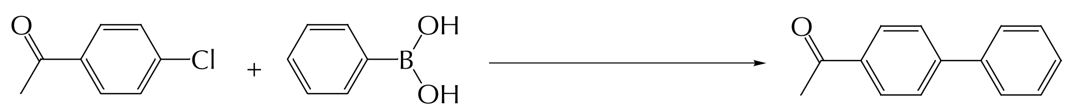
f. FibreCat®

FibreCat® catalysts, which are immobilized precious metal catalysts with ligands, use graft polymerized polyethylene resins in the form of fibres as a carrier. Their fibre formation allow functional groups to easily interact with substances.
The catalysts are readily recoverable and reusable just as with common immobilized catalysts. Other excellent features are listed below.

[Features]

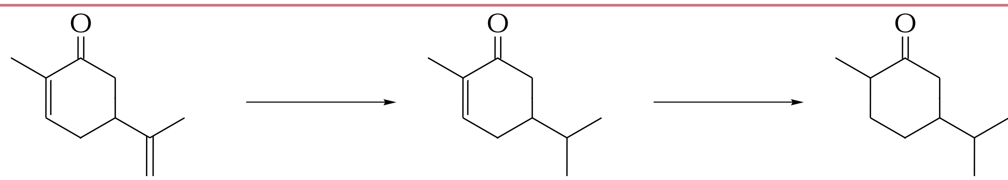
1. **Easy filtration → FibreCat® can be easily separated by filtration because they are insoluble in almost all solvents.**
2. **High safety → FibreCat® can be treated as noncombustible heterogeneous catalysts.**
3. **High mechanical strength → FibreCat® are easy to handle because they suffer no physical degradation on stirring.**
4. **Wide compatibility → FibreCat® can be used in a wide range of solvents, such as aqueous, organic, and even nonpolar hydrocarbon.**
5. **High heat stability → FibreCat® can be used in general at 120℃ and up to 150℃ depending on reaction conditions.**
6. **Ligand addition → FibreCat® do not require additional ligands because they already contain ligands.**

[Reaction 1] 1000 Series： Pd containing catalysts (for use in coupling reactions such as Suzuki coupling reaction, Hech reaction)



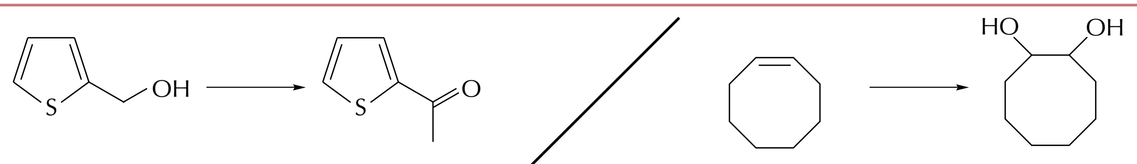
condition : KF, 1mol% Pd, toluene, 90℃, 24h

[Reaction 2] 2000 Series： Rh containing catalysts (for use in hydrogenation reactions)



condition : H_2, 1mol% Rh, THF, 3bar, 70℃, 2h

[Reaction 3] 3000 Series： Ru or Os containing catalysts (for use in selective oxidation reactions)



condition : 10mol% Ru, toluene, 3bar, 60℃, 6h
condition : 0.1mol% Os, $(CH_3)_3N \rightarrow O$, 60℃, 2h

Wako Catalog No.	description	Remark	Pkg. Size	
324-56621 320-56623	**FibreCat® 1001** (Palladium(II) Acetate-Triphenylphosphine PE fibres)	Pd content： 2.7~4%	1g 5g	Pd
321-56631 327-56633	**FibreCat® 1007** (Palladium(II) Acetate-Dicyclohexylphenylphosphine PE fibres)	Pd content： 3~4%	1g 5g	
328-56641 324-56643	**FibreCat® 1026** (Acetonitrile Dichloropalladium(II)-Triphenylphosphine PE fibres)	Pd content： 3.5~4.5%	1g 5g	
325-56651 321-56653	**FibreCat® 2003** (Bis(η-norbornadiene)rhodium(I) Tetrafluoroborate-Triphenylphosphine PE fibres)	Rh content： 3~4%	1g 5g	Rh
322-56661 328-56663	**FibreCat® 2006** (Di-μ-chlorobis(η-norbornadiene)dirhodium(I)-Triphenylphosphine PE fibres)	Rh content： 2~3.5%	1g 5g	
329-56671 325-56673	**FibreCat® 3002** (Sodium Ruthenate(VII)-Triethylamine PE fibres)	Ruc content： 3.5~4.5%	1g 5g	Ru
326-56681 322-56683	**FibreCat® 3003** (Osmium(VIII) Oxide-Pyridine PE fibres)	Osc content： Approx. 7.5%	1g 5g	Os
323-56691 329-56693	**FibreCat® 3004** (Dipotassium Dioxotetrahydroxoosmate(VIII)-Triethylamine PE fibres)	Os content： Approx. 7.5%	1g 5g	
326-56701 322-56703	**FibreCat® 4001** (Dihydrogen Hexachloroplatinate(IV) n-Hydrate-Pyridine PE fibres)	Pt content： 3.5~5%	1g 5g	Pt
323-56711 329-56713	**FibreCat® 4003** (*cis*-Bis(acetonitrile)dichloroplatinum(II)-Triphenylphosphine PE fibres)	Pt content： 3.5~4.5%	1g 5g	

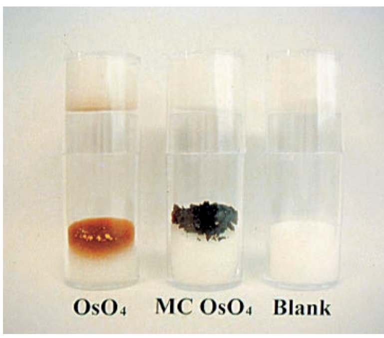
g. Custom synthesis using Microencapsulated Osmium (VIII) oxide (OsO_4)

Wako offers custom synthesis of (chiral) diol on an industrial scale using Microencapsulated Osmium (VIII) OsO_4, which has less volatility.

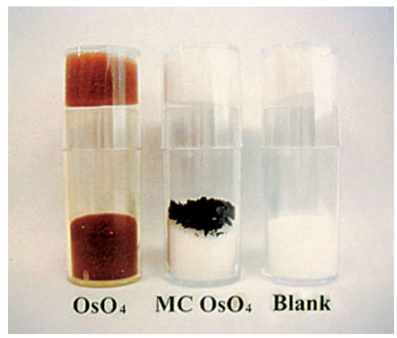
[Features]

1. **We offer custom synthesis on an industrial scale using Microencapsulated Osmium (VIII) OsO_4.**
2. **Residual OsO_4 in synthetic products is fewer than when simple OsO_4 is used. (Actual concentration of residual OsO_4 is reported)**
3. **Microencapsulated Osmium (VIII) OsO_4 can be designed for various reaction systems.**
4. **Microencapsulated Osmium (VIII) OsO_4 can be used for asymmetric reaction with asymmetric ligands.**

Low Toxicity due to Low Volatility



[Before]



[After 3 days]

C. Reaction in water

a. Rare-earth Triflate

It is important for green chemistry that auxiliary substances in organic reactions are used as little as possible and that they are innocuous when used.
Solvents are usually mostly used in organic reactions.
If the reactions can be conducted in aqueous solution instead of organic solvents which are commonly used, the environmental impacts are significantly reduced. Rare-earth triflates, as Lewis acid catalysts, allow the use aqueous solution as a solvent for a wide range of organic reactions.

Catalog No.	Description	MW	CAS	Package Size
195-11391 191-11393	**Scandium (III) Trifluoromethanesulfonate**	$(CF_3SO_3)_3Sc$=492.16	144026-79-9	1 g 5 g

Sc

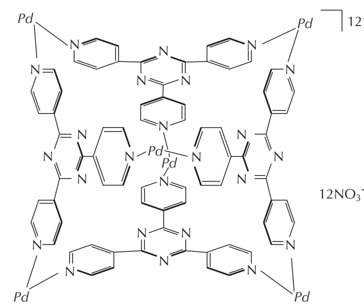
[References]
1) S. Kobayashi, I. Hachiya, M. Araki and H. Ishitani: *Tetrahedron Lett.*, **34**, 3755 (1993).
2) S. Kobayashi : *Synlett*, 689 (1994).

Palladium-Nanocage

Pd

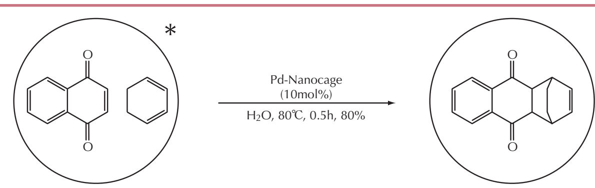
Cat. #160-20471 **1 g**
RT, Solid (pale yellow powder)
MW : 2992.48 ($C_{84}H_{96}F_{48}O_{36}Pd_6$)

Pd-Nanocage which is self-assembled from(en)Pd$(NO_3)_2$ (as an adhesive) and triazine derivatives (as a molecular panel) is found to promote the aerobic, aqueous oxidation of styrene and its derivatives. It acts as a reverse phase-transfer catalyst, whereas (en)Pd^{2+} as an oxidation catalyst.

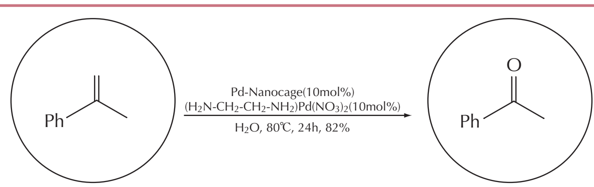


Pd=(H_2N-CH_2-CH_2-NH_2)Pd
Pd-Nanocage

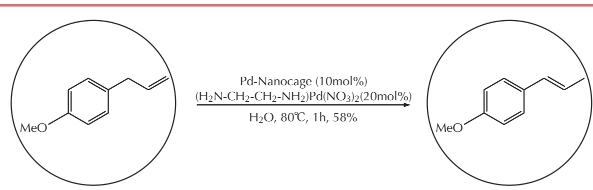
[Reaction 1]
Diels-Alder Reaction



[Reaction 2]
Wacker Oxidation Reaction



[Reaction 3]
Wacker Oxidation Reaction



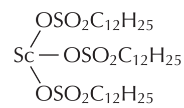
** : ○ shown in the above-mentioned chemical formulae shows the space of Nanocage.

[References]
1) D. Oguro, M. Miyazawa, H. Oka, K. Yamaguchi, K. Ogura, M.Fujita : *Nature*, **378**, 469 (1995).
2) T.Kusukawa, M. Fujita : *J. Am. Chem. Soc.*, **121**, 1397 (1999).
3) H. Ito, T. Kusukawa, M. Fujita : *Chem. Lett.*, 598 (2000).
4) M. Yoshizawa, T. Kusukawa, K. Yamaguchi, M. Fujita : *J. Am. Chem. Soc.*, **122**, 6311 (2000).

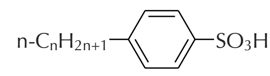
Catalysts

b. Surfactant Combined Catalysts

Organic reactions in water without use of damaging organic solvents are of great current interest from the standpoint of environmental or ecological concerns. The noble catalysts, which enable the use of water as a solvent for a wide range of organic reactions, will contribute to progress in environmentally benign chemical processes by reducing the use of organic solvents.



STDS



DBSA (n=10-14)

[Features] Immediately forming reaction substrates and emulsion in the water, realizing high-level hydrophobic condition

Sc Scandium Tris (dodecyl sulfate) Trihydrate [STDS]

Cat. #194-12341 1 g
#190-12343 5 g

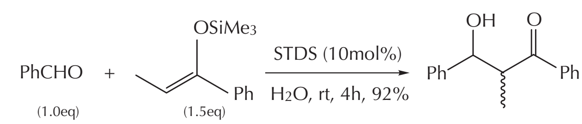
RT, Solid (white, powder)

MW : 895.17 ($Sc(OSO_3C_{12}H_{25})_3 \cdot 3H_2O$)

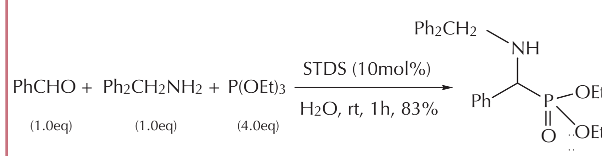
Assay (Titration) : 95.0+ %

STDS is a Lewis acid-surfactant combined compound. The newly designed Lewis acid has been shown to be a noble catalyst in the aldol reactions, Mannich-type reactions, allylation reactions and Michael reactions. It has very different characteristics from conventional Lewis acids. The stable dispersion systems including the catalyst and organic substrates are formed in water and highly water-sensitive ketene silyl acetals even react smoothly in the dispersion systems.

[Reaction 1] Aldol Reaction



[Reaction 2] Synthesis of α-Amino phosphonic acid



linear-Alkylbenzenesulfonic Acid [DBSA]

Cat. #017-15065 500 mL

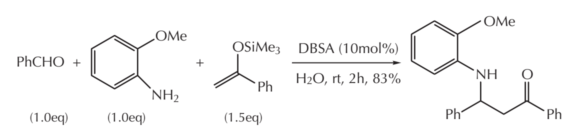
~25℃, protect from light, Liquid

[Class 8, UN 2584]

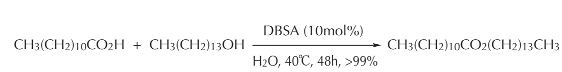
MW : 326.49 ($C_{18}H_{30}O_3S$)

DBSA is a Brønsted acid-surfactant combined compound and catalyzes efficiently three-components Mannich-type reaction of aldehydes, amines and silyl enolates in water. DBSA with the organic substrates forms stable colloidal particles in water and dehydrate reactions like an esterification can even be achieved in the colloidal particles.

[Reaction 1] Mannich Reaction



[Reaction 2] Esterification



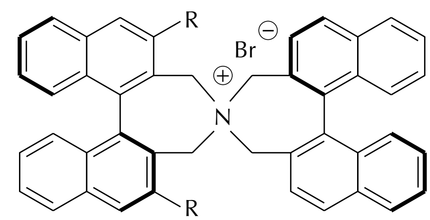
[References]

1) K. Manabe, Y. Mori, T. Wakabayashi, S. Nagayama and S. Kobayashi : *J. Am. Chem. Soc.*, **122**, 7202 (2000).
2) T. Wakabayashi, S. Kobayashi : *Tetrahedron Lett.*, **39**, 5389 (1998).
3) K. Manabe, Y. Mori and S. Kobayashi : *Synlett.*, **9**, 1401 (1999).
4) K. Manabe, X-M. Mori, S. Kobayashi : *J. Am. Chem. Soc.*, **123**, 10101 (2001).

D. Chiral Phase-Transfer Catalysts(PTC)

a. Maruoka Catalysts

Chiral phase-transfer catalysts, which are optically active spiro ammonium salts with 2 binaphthyl rings that allow for easy molecule designing, were invented by Professor Maruoka of the Kyoto University.
(R,R)-3,4,5-Trifluorophenyl-NAS Bromide shows high catalytic activity and high enantioselectivity in asymmetric alkylation of α-amino acid derivatives[1).
By using (R,R)-3,5-Bistrifluoromethylphenyl-NAS Bromide, β- hydroxy α-amino acid derivatives which are important chiral units of biologically active peptides are obtained in good yield by the aldol reaction of glycine derivatives with aldehydes. It was found that erythro isomers, which are major products, were obtained with high enantioselectivity[2).



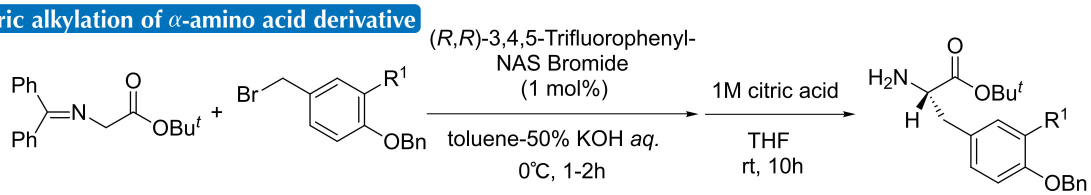
① (R=3,5-Bistrifluoro-Ph), ② (R=3,4,5-F^3-Ph)

[Features]

1. **The rational molecular design of C_2-symmetric chiral quaternary ammonium salt**
2. **Reaction under mild organic / aqueous biphase conditions**
3. **Catalyzing the asymmetric alkylations of α-amino acid derivatives and the direct asymmetric aldol reactions of glycine schiff base with aldehydes to the corresponding β-hydroxy-α-amino acid derivatives**

[Reaction 1]

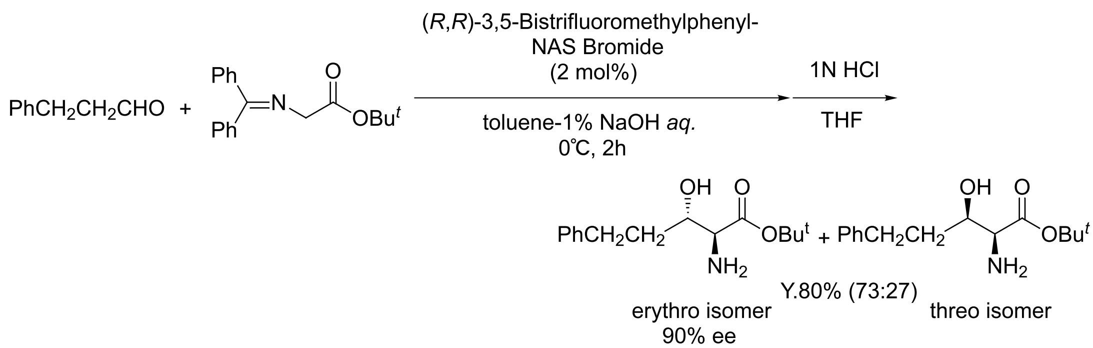
Asymmetric alkylation of α-amino acid derivative



R^1=OBn : Y. 81%, 98% ee
R^1=H : Y. 83%, 98% ee

[Reaction 2]

Asymmetric aldol reaction of β-hydroxy-α-amino acid derivative



Maruoka Catalysts

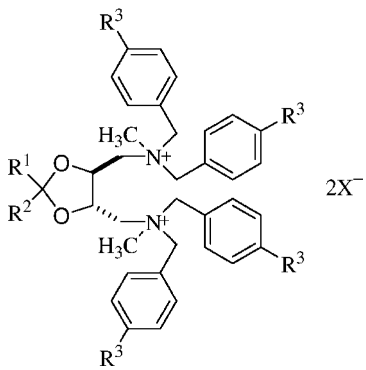
Wako Catalog No.	Description	Package Size	Storage
029-14921	***(R,R)*-3,5-Bistrifluoromethylphenyl-NAS Bromide**	100 mg	RT
025-14923	[Maruoka Catalyst RR-Bistrifluoromethylphenyl Br Form]	500 mg	
201-15921	***(R,R)*-3,4,5-Trifluorophenyl-NAS Bromide**	100 mg	RT
207-15923	[Maruoka Catalyst RR-Trifluorophenyl Br Form]	500 mg	

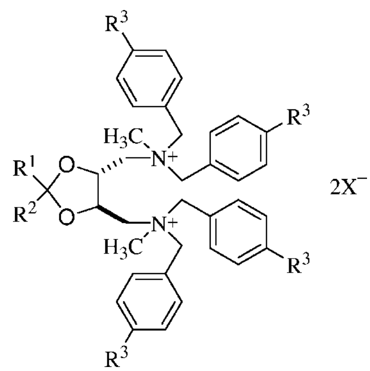
[References]

1) Ooi T., Takeuchi M., Kameda M. and Maruoka K. : *J. Am. Chem. Soc.*, **122** (21), 5228-5229 (2000).
2) Ooi T., Taniguchi M., Kameda M., Maruoka K. : *Angew. Chem. Int.*

b. TaDiAS

TaDiAS (Tartrate-derived DiAmmonium Salt), an asymmetric phase-transfer catalyst which contains 2 quarternary ammonium salts in its molecule, was developed by the Shibasaki group of The University of Tokyo.
(It is considered that) TaDiAS is believed to produce high asymmetric yields of asymmetric compounds by fixation of the anion position in asymmetric space as a result of the collaboration of 2 quarternary ammonium salts.
TaDiAS 1a and 1b, the iodide salts, show high catalytic activity and high enantioselectivity in asymmetric alkylation of α-amino acid derivatives. TaDiAS 2a and 2b, the tetrafluoroboric acids, show high catalytic activity and high enantioselectivity in asymmetric Michael addition of α-amino acid derivatives[1) 2)]



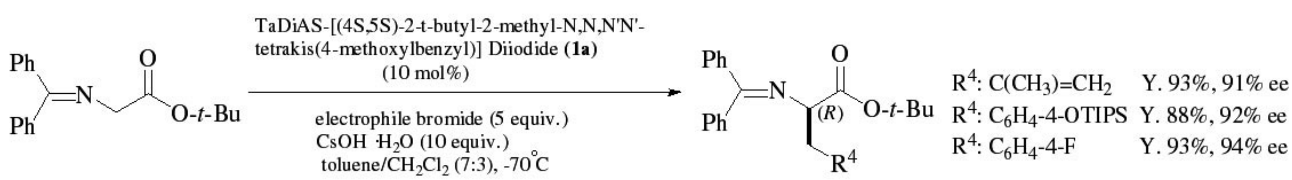


1a) R^1: t-Bu, R^2: Me, R^3: OMe, X: I
TaDiAS-[(4S,5S)-2-t-butyl-2-methyl-N,N,N'N'-tetrakis(4-methoxylbenzyl)] Diiodide
2a) R^1: Pr, R^2: Pr, R^3: Me, X: BF_4
TaDiAS-[(4S,5S)-2,2-dipropyl-N,N,N',N'-tetrakis(4-methylbenzyl)] Bis(tetrafluoroborate)

1b) R^1: t-Bu, R^2: Me, R^3: OMe, X: I
TaDiAS-[(4R,5R)-2-t-butyl-2-methyl-N,N,N'N'-tetrakis(4-methoxylbenzyl)] Diiodide
2b) R^1: Pr, R^2: Pr, R^3: Me, X: BF_4
TaDiAS-[(4R,5R)-2,2-dipropyl-N,N,N',N'-tetrakis(4-methylbenzyl)] Bis(tetrafluoroborate)

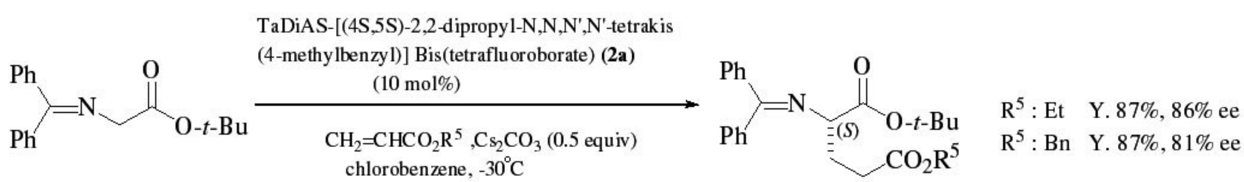
[Reaction 1]

Asymmetric alkylation of α-amino acid derivative[1) 2)]



[Reaction 2]

Asymmetric Michael addition of α-amino acid derivatives[1) 2)]



TaDiAS

Catalog No.	Description	Pkg. Size
208-16151	TaDiAS-[(4S,5S)-2-t-butyl-2-methyl-N,N,N'N'-tetrakis(4-methoxylbenzyl)] Diiodide	100 mg
201-16141	TaDiAS-[(4R,5R)-2-t-butyl-2-methyl-N,N,N'N'-tetrakis(4-methoxylbenzyl)] Diiodide	100 mg
202-16171	TaDiAS-[(4S,5S)-2,2-dipropyl-N,N,N',N'-tetrakis(4-methylbenzyl)] Bis(tetrafluoroborate)	100 mg
205-16161	TaDiAS-[(4R,5R)-2,2-dipropyl-N,N,N',N'-tetrakis(4-methylbenzyl)] Bis(tetrafluoroborate)	100 mg

[References]

1) T. Shibuguchi, Y. Fukuta, Y. Akachi, A. Sekine, T. Ohshima, M. Shibasaki : *Tetrahedron Lett.*, **43**, 9539 (2002).
2) T. Ohshima, V. Gnanadesikan, T. Shibuguchi, Y. Fukuta, T. Nemoto, M. Shibasaki : *J. Am. Chem. Soc.*, **125**, 11206 (2003).

Various Kinds of Compounds for Organic Chemistry

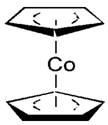
These three kinds of brochures are available now!!!

F. Metallocene

Metallocene is a general term for bis(cyclopentadienyl) complex salts, $(C_5H_5)_2M$, consisting of 2 cyclopentadiene rings and various transition metals. Ferrocene is the prototypical metallocene. It has high heat stability and many derivatives are produced.

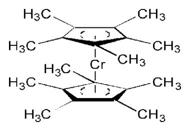
In addition to the ferrocene, many metallocenes containing various transition metals such as Hf, Ru and Ti have been developed and are on the market. Wako offers the following metallocenes and its derivatives.

Cobaltocene



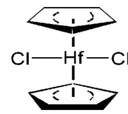
[1277-43-6]
320-39241 1g
320-39241 1g
320-39241 1g

Bis(pentamethylcyclopentadienyl)-chromium(II)



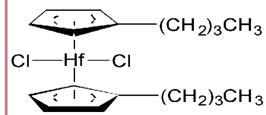
[74507-61-2]
323-27881 1g

Bis(cyclopentadienyl)-hafnium(IV) Dichloride



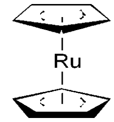
[12116-66-4]
029-13201 1g

Bis(butylcyclopentadienyl) hafnium(IV) Dichloride



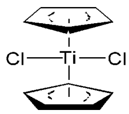
[85722-08-3]
324-39261 500mg

Ruthenocene



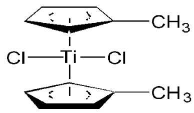
[1287-13-4]
327-39251 1g

Bis(cyclopentadienyl)titanium Dichloride



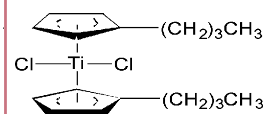
[1271-19-8]
204-04281 1g
200-04283 10g
202-04282 25g

Bis(methylcyclopentadienyl)-titanium(IV) Dichloride



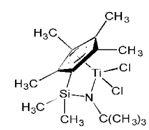
[1282-40-2]
324-35121 1g

Bis(butylcyclopentadienyl) titanium(IV) Dichloride



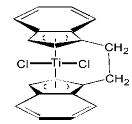
[1282-40-2]
321-27963 5g

Bis(*t*-butylcyclopentadienyl)-titanium(IV) Dichloride



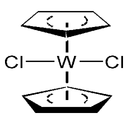
[79269-71-9]
327-27921 1g
323-27923 5g

Ethylenebis(1-indenyl)-titanium(IV) Dichloride



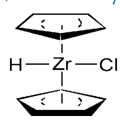
[112531-76-7]
327-39371 500mg

Bis(cyclopentadienyl)-tungsten(IV) Dichloride



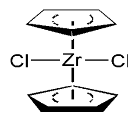
[12184-26-8]
322-52401 1g

Bis(cyclopentadienyl)zirconium(IV) Chloride Hydride



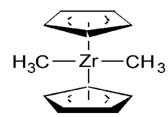
[37342-97-5]
327-35111 5g
321-39271 500mg
325-35112 25g

Zirconocene Dichloride



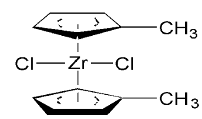
[1291-32-3]
260-00541 1g
266-00543 5g
268-00542 25g

Bis(cyclopentadienyl)-dimethyl-zirconium(IV)



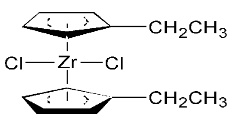
[12636-72-5]
320-28011 1g
326-28013 5g

Bis(methylcyclopentadienyl)-zirconium(IV) Dichloride



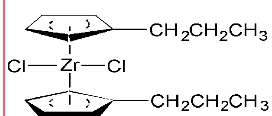
[12109-71-6]
328-39301 500mg

Bis(ethylcyclopentadienyl)-zirconium(IV) Dichloride



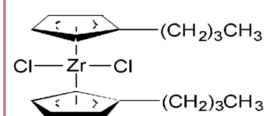
[73364-08-6]
325-39291 500mg

Bis(propylcyclopentadienyl) zirconium(IV) Dichloride



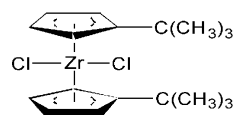
[73364-09-7]
325-39311 500mg

Bis(*n*-butylcyclopentadienyl)-zirconium(IV) Dichloride



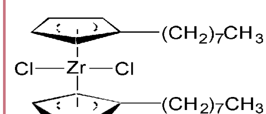
[73364-10-0]
324-27931 1g
320-27933 5g

Bis(*t*-butylcyclopentadienyl)-zirconium(IV) Dichloride



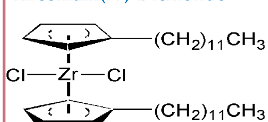
[32876-92-9]
328-27951 1g
324-27953 5g

Bis(octylcyclopentadienyl)-zirconium(IV) Dichloride



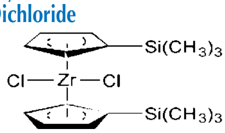
[191803-21-1]
322-45051 100mg

Bis(dodecylcyclopentadienyl)-zirconium(IV) Dichloride



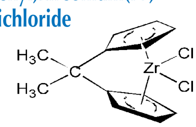
[191803-23-3]
328-39281 500mg

Bis(trimethylsilylcyclopentadienyl)zirconium(IV) Dichloride



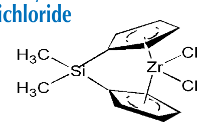
[60938-59-2]
322-39321 500mg

Isopropylidenebis(cyclopentadienyl)zirconium(IV) Dichloride



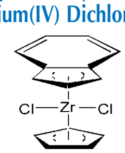
[138533-79-6]
321-47341 100mg

Dimethylsilylenebis(cyclopentadienyl)zirconium(IV) Dichloride



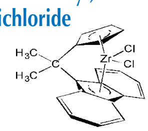
[86050-32-0]
321-39391 500mg

(Cyclopentadienyl)(indenyl)-zirconium(IV) Dichloride



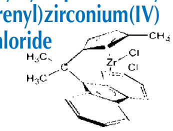
[80155-52-8]
329-39331 500mg

Isopropylidene(cyclopentadienyl)-(9-fluorenyl)zirconium-(IV) Dichloride



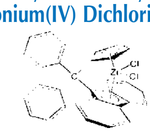
[130638-44-7]
328-47351 100mg

Isopropylidene(3-methylcyclopenta-dienyl)(9-fluorenyl)zirconium(IV) Dichloride



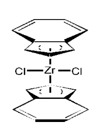
[133190-48-4]
325-47361 100mg

Diphenylmethylidene(cyclopentadienyl)(9-fluorenyl)-zirconium(IV) Dichloride



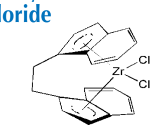
[132510-07-7]
320-39361 500mg

Bis(indenyl)zirconium(IV) Dichloride

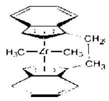
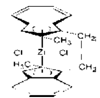
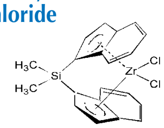
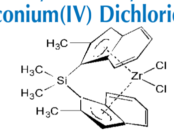
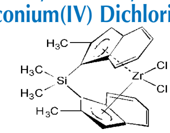
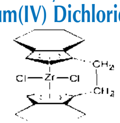
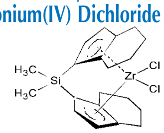


[12148-49-1]
321-49041 1g
327-49043 5g

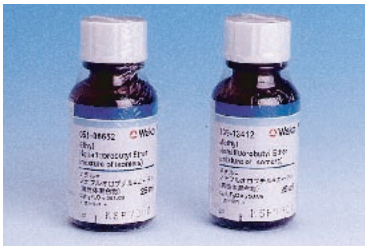
***meso*-Ethylenebis-(1-indenyl)-zirconium(IV) Dichloride**



[162429-20-1]
329-45061 100mg

***rac*-Ethylenebis-(1-indenyl)-dimethylzirconium(IV)**  [136844-77-4] 326-45071 100mg 322-45073 500mg	***rac*-1,2-Ethylenebis(2-methyl-1-indenyl)-zirconium(IV) Dichloride**  [143278-87-9] 326-39461 100mg	***rac*-Dimethylsilylenebis-(1-indenyl)-zirconium(IV) Dichloride**  [121009-93-6] 324-39401 500mg	***meso*-Dimethylsilylenebis-(2-methyl-1-indenyl)-zirconium(IV) Dichloride**  [182210-68-0] 323-45081 100mg	***rac*-Dimethylsilylenebis-(2-methyl-1-indenyl)-zirconium(IV) Dichloride**  [149342-08-5] 320-45091 100mg
***rac*-Ethylenebis-(4,5,6,7-tetrahydro-1-indenyl)-zirconium(IV) Dichloride**  [100163-29-9] 324-39381 100mg 320-39383 500mg	***rac*-Dimethylsilylenebis-(4,5,6,7-tetrahydro-1-indenyl)-zirconium(IV) Dichloride**  [126642-97-5] 322-47371 100mg			

CFC-Alternatives



Non-ozone-depleting fluorinated solvents were developed as Chlorofluorocarbon (CFC) -alternatives.
Global warming potential is low. We offer two mixtures of *n*- and iso-butyl isomers, 99.0+% (cGC)

Ethyl Nonafluorobutyl Ether (mixture of isomers), 99.0+% (cGC)

051-06652 25mL
055-06655 500mL
RT, Liquid
M W : 264.09 ($C_6H_5F_9O$)

Methyl Nonafluorobutyl Ether (mixture of Isomers), 99.0+% (cGC)

139-13412 25mL
133-13415 500mL
RT, Liquid
M W.: 250.06 ($C_5H_3F_9O$)

[Features]
1. **Water-repellent**
2. **Easily soluble in various organic solvents**

n-butyl isomer *iso*-butyl isomer
R=methyl or ethyl

[Solubility in various organic solvents at 25℃]

	Solvent					
	Methanol	1-Butanol	Hexane	Dodecane	Diethylether	Acetone
Ethyl Nonafluorobutyl Ether	○	○	○	○	○	○
Methyl Nonafluorobutyl Ether	○	16.8 (w/w%)	○	5.9 (w/w%)	○	○

○ : Very soluble in the solvent.

☞ See page #26 on the related products.

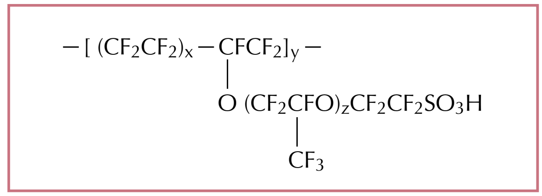
Catalysts

G. Solid Super-Acid Catalysts

Nafion® NR-50

RT, Solid (nearly white~brown, pellet)

Strong acid polymer in a bead form (perfluorinated ion exchange polymer : PFIEP), which can be applied for a wide range of organic reactions requiring an acid catalysis.



Nafion® is a registered mark of DuPont.

[How to recycle]

1. Boil for two hours in 150 mL of H_2O, followed by filtration
2. Add the resin that is obtained after filtration to 200 mL of 20~25% nitric acid, and agitate it for 4~5 hours at a room temperature. Then filter it.
3. Repeat nitric acid treatment several times.
4. Rinse the resin that has been treated with acid with distilled water until the water becomes neutral.
5. Dry the rinsed resin for 24 hours at a temperature of 100~105℃.

Catalog No.	Description	CAS	Package Size
144-05991 142-05992	Nafion® NR-50	118473-68-0	5g 25g

[References]

1) S. Kanemoto, H. Saimoto, K. Oshima, H. Nozaki : *Tetrahedron Lett.*, **25**, 3317 (1984).
2) M. Hino, K. Arata : *Apple. Catol.*, **18**, 401 (1985).

Nafion® Dispersion Solution

Nafion®is a Perfluorosulfonic Acid/PTFE copolymer in the (H^+) form and has unique characters that are widely applicable. Three wt. % formulations, 5%, 10% and 20% are available.

[Physical properties]

	DE520	DE521	DE1020	DE1021	DE2020	DE2021
Polymer Content (%)	min.5.0 max.5.4	min.5.0 max.5.4	min.10.0 max.12.0	min.10.0 max.12.0	min.20.0 max.22.0	min.20.0 max.22.0
Water Content (%)	45±3	45±3	87~90	87~90	34±2	34±2
VOC Content (%)	50±3	50±3	< 1	< 1	46±2	46±2
1-Propanol	48±3	48±3	—	—	44±2	44±2
Ethanol	< 4	< 4	—	—	< 2	< 2
Mixed Ethers and Other VOCs	< 1	< 1	—	—	< 1	< 1
Specific Gravity	0.92-0.94	0.92-0.94	1.05-1.07	1.05-1.07	0.97-0.99	0.97-0.99
Available Acid Capacity (meq/g, H^+ polymer basis)	> 1.00	> 0.90	> 1.00	> 0.90	> 1.00	> 0.90
Viscosity (cP;at 25℃ and 40sec^{-1} Shear Rate)	10~20	10~20	3~6	3~6	200~400	200~400

Wako Catalog No.	Description	Package Size	Storage
326-46372 324-46373	5% Nafion® Dispersion Solution DE521	25 mL 100mL	RT
323-46382 321-46383	5% Nafion® Dispersion Solution DE520	25 mL 100 mL	
320-46392 328-46393	10% Nafion® Dispersion Solution DE1021	25 mL 100 mL	
323-46402 321-46403	10% Nafion® Dispersion Solution DE1020	25 mL 100 mL	
320-46412 328-46413	20% Nafion® Dispersion Solution DE2021	25 mL 100 mL	
327-46422 325-46423	20% Nafion® Dispersion Solution DE2020	25 mL 100 mL	

Zirconia, Sulfated (SO_4 / ZrO_2)
Zirconia Tungstate (WO_3 / ZrO_2)

Zirconia, Sulfated and Zirconia Tungstate are solid yet have higher acid strength than sulfate and exhibit excellent catalytic efficiency in various acid catalysis reactions such as esterification, acylation[1], isomerization, ether synthesis, alkylation, disproportionation, polymerization and degradation.

These products can be used at relatively high reaction temperature. Since these can be treated as solids, the corrosivity for apparatuses is low and the acid treatment is rarely required. Therefore these are superior catalysts from the perspective of environmental protection. Wako provides 2 types of products, powder and pellet, for use at various reaction conditions

[Example reactions]

Esterification

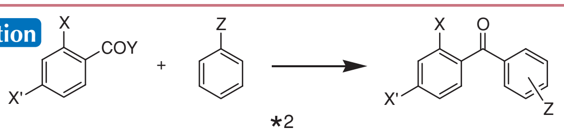
$$R^1\text{-COOH} \quad + \quad R^2\text{-OH} \longrightarrow R^1\text{-COOR}^2$$

(1) R^1 = methyl, R^2 = methyl, ethyl, propyl, butyl
(2) R^1 = acryl, R^2 = ethyl
(3) R^1 = salicyl, R^2 = methyl
(4) R^1 = phthal, terephthal, R^2 = octyl, 2-ethylhexyl
(5) R^1 = methacryl, R^2 = methyl *1
(6) R^1 = heptyl, R^2 = methyl

*1

Temp.	130 ℃
Methanol/Methacrylic Acid	3 mol/mol
LHSV	1 h^{-1}
Methacrylic Acid Conv.	99
Methyl Methacrylate Select.	>99

Acylation



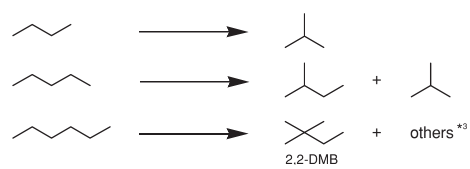
(1) X = H, X' = H, Y = Cl, Z = CH_3
(2) X = H, X' = H, Y = OOCPh, Z = CH_3
(3) X = H, X' = H, Y = OH, Z = CH_3
(4) X = Cl, X' = H, Y = Cl, Z = CH_3
(5) X = Cl, X' = H, Y = Cl, Z = Cl
(6) X = H, X' = Cl, Y = Cl, Z = Cl *2

*2

Catalyst	Yield (%)
SO_4/ZrO_2	31.7
WO_3/ZrO_2	10.5
$SiO_2\text{-}Al_2O_3$	<0.1

Reaction condition: *p*-chlorobenzoyl chloride 20 mmol, Chlorobenzene 200 mmol, Catalyst 2.0 g, Temp.135℃, Time 3 h

Isomerization



*3

Catalyst	Conversion (wt %)	2,2-DMB/Σ C6 (wt %)
Pt-SO4/ZrO2 (Pellet)	89.8	27.5
Pt-SO4/ZrO2 (Powder)	84.5	15.6

Reaction condition: Temp. 200℃, Press. 1.08 Mpa, LHSV 1.5 h^{-1}, H_2/Hexane 5 mol/mol

Synthesis of ether by dehydration

MeOH (flow rate 1.5g/h)
N_2 (flow rate 100mL /min)
Column: WO_3/ZrO_2, Pellet 1.0g

$$CH_3OH \xrightarrow[200℃]{} CH_3OCH_3$$

DME

Activity at just after start of reaction (GC) DME/(DME+MeOH): 50%
Activity at 20 hours after start of reaction (GC) DME/(DME+MeOH): 40%

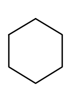
Alkylation

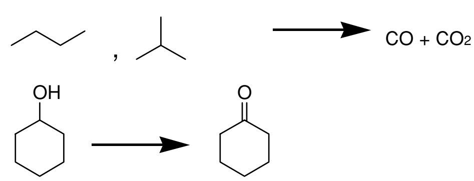
isobutane + butenes → $C_5\text{-}C_{11}$ alkylates

xylenes + styrene → phenylxylylethane

Others

1-octene, 1-decene, β-pinene → dimer, trimer

 + heptane → jet fuel



Just before use, this product should be left to dry for 1 hour at an air temperature of 300 ~ 500℃ to obtain sufficient expression activity.

[References]

1) Matsuzawa, K. : *Prepr. Am. Chem. Soc. Div. Pet. Chem.*, **42**(4), 734 (1997).

Wako Catalog No	Description	Grade	Package Size
269-01471	Zirconia, Sulfated	Wako 1st Grade	5 g
267-01472			25 g
268-01762	Zirconia, Sulfated, Pellet	for Organic Synthesis	25 g
260-01761			100 g
267-01771	Zirconia Tungstate	Wako 1st Grade	5 g
265-01772			25 g
262-01782	Zirconia Tungstate, Pellet	for Organic Synthesis	25 g
264-01781			100 g

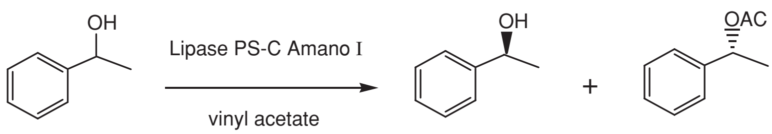
H. Biocatalysts

Catalysts for Asymmetric Synthesis

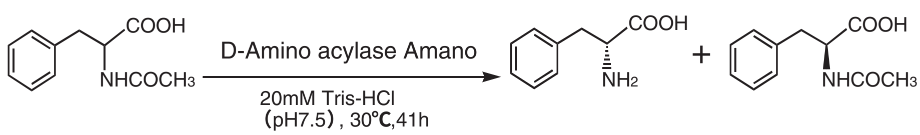
Amano Lipase / Acylase

Utilization of the biocatalysts that are enzymes or microorganisms is a notable technique from the green chemical standpoint of "preventing waste". It has been recognized as being useful for obtaining optically active substances without difficulty. Wako offers hydrolases such as lipase and acylase for producing optically active substance. Three kinds of reusable immobilized enzymes are also in our lineup.

[Reaction 1]



[Reaction 2]



1.0M *N*-Ac-D,L-Phe D-Phe *N*-Ac-L-Phe

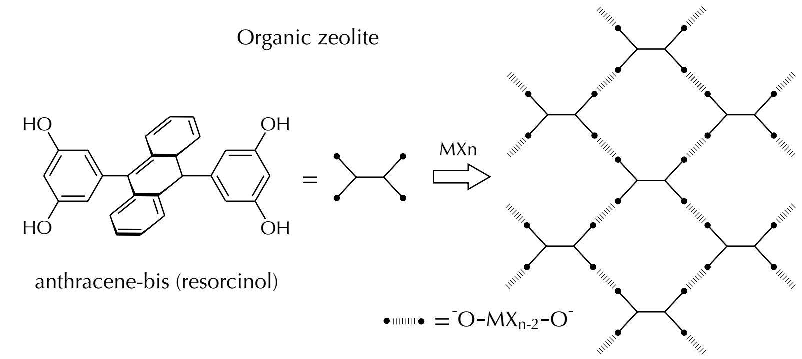
Wako Catalog No.	Description	Source	Activity	Optimum pH	Optimum Temperature	Package Size
321-58331 327-58333	Lipase AS Amano	*Aspergillus niger*	not less than 12,000units/g	6.5	45℃	10 g 50 g
328-58341 324-58343	Lipase M Amano 10	*Mucor javanicus*	not less than 10,000units/g	7.0	35℃	10 g 50 g
325-58351 321-58353	Lipase F-AP15	*Rhizopus oryzae*	not less than 150,000units/g	7.0	40℃	10 g 50 g
322-58361 328-58363	Lipase G Amano 50	*Penicillium camembertii*	not less than 50,000units/g	5.0	40℃	10 g 50 g
329-58371 325-58373	Lipase AYS Amano	*Candida rugosa*	not less than 30,000units/g	7.0	45℃	10 g 50 g
326-58381 322-58383	Lipase PS Amano	*Burkholderia cepacia*	not less than 30,000units/g	7.0~8.0	45℃	10 g 50 g
323-58391 329-58393	Lipase AK Amano	*Pseudomonas fluorescence*	not less than 25,000units/g	8.0	60℃	10 g 50 g
326-58401 324-58402	Lipase PS-C Amano I, Immobilized on Ceramic	*Burkholderia cepacia*	not less than 1,000units/g	7.0~8.0	45℃	5 g 25 g
323-58411 321-58412	Lipase PS-C Amano II, Immobilized on Ceramic	*Burkholderia cepacia*	not less than 600units/g	7.0~8.0	45℃	5 g 25g
320-58421 328-58422	Lipase PS-D Amano I, Immobilized on Diatomaceous Earth	*Burkholderia cepacia*	not less than 500units/g	7.0~8.0	45℃	5 g 25 g
327-58431 323-58433	Acylase Amano	*Aspergillus melleus*	not less than 30,000units/g	8.0	50℃	10 g 50 g
329-61061 325-61063	D-Aminoacylase Amano	*Escherichia coli*	not less than 10.1Munits/g	8.0	45℃	10 Munits 50 Munits

I. Anthracene

Organic zeolite is formed from anthracenebis- resorcinol with metal alkoxides such as La(OPr-*i*)$_3$ or Zr(OBu-*t*)$_4$ based on hydrogen-bonded network. Pd-Nanocage is self-assembled from (en)Pd(NO_3)$_2$ (as an adhesive) and triazine derivatives (as a molecular panel). These unique materials are usefully effective catalysts for the organic reactions in aqueous medium. For example, the Diels-Alder reaction, Wacker oxidation, Michael reaction and nitroaldol reaction are accelerated in water by using these catalysts. These catalytic performances are caused by the function of metal and hydrophobic space containing their inner.

Anthracene-9,10-bis (5-resorcinol)

Cat. #018-18851 100 m g
#014-18853 1 g
RT, Solid (pale yellow, powder)
MW : 394.42 ($C_{26}H_{18}O_4$)
CAS : 153715-08-3
Assay (HPLC) : 98.0 + %



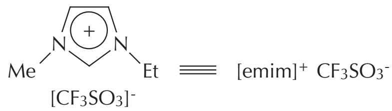
J. Ionic Liquid

Ionic liquid is quaternary ammonium salt with low melting point and high boiling point. With its high stability at from high to low temperatures.

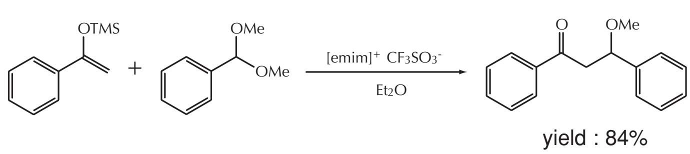
[Features]
1. **Nonvolatile liquid**
2. **Having low-viscosity although the liquid is ionic**
3. **Heat resistant and with wide liquid temperature range**
4. **Easy to collect and recycle because of its non-miscibility with organic solvent**

1-Ethyl-3-methylimidazolium Trifluoromethanesulfonate

Cat. #059-07111 10 g
RT, Liquid (pale brown, liquid)
MW : 260.23 ($C_7H_{11}F_3N_2O_3S$)
CAS : 145022-44-2



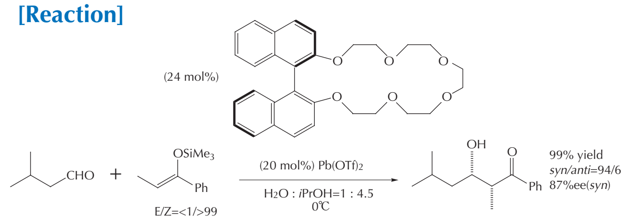
[Reaction 1] Aldol Reaction



A. Chiral Crown Ethers

Chiral crown ether, which forms complexes with free metals in water solution, has turned out to be an effective asymmetric ligand for catalytic asymmetric synthesis reaction.

[Reaction]

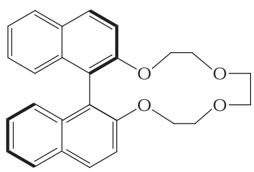


[References]

S. Nagayama, S. and S. Kobayashi : *J. Am. Chem. Soc.*, **122**, 11531 (2000).

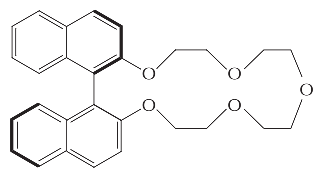
(*R*)-2,2'-Binaphthyl-14-crwon-4

Cat. #023-14681 1 g
RT, Solid (pale brown, powder)
CAS : 128778-82-5



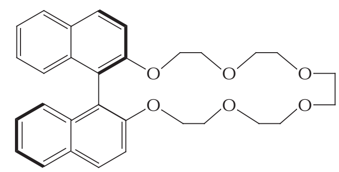
(*R*)-2,2'-Binaphthyl-17-crwon-5

Cat. #023-14701 1 g
RT, Solid (pale yellow, powder)
CAS : 99630-51-0



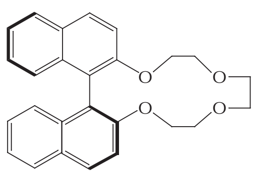
(*R*)-2,2'-Binaphthyl-20-crwon-6

Cat. #027-14721 1 g
RT, Solid (colorless, viscous mass)
CAS : 75684-69-4



(*S*)-2,2'-Binaphthyl-14-crwon-4

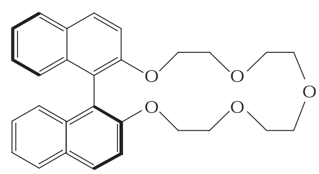
Cat. #020-14691 1 g
RT, Solid (pale yellow, powder)
CAS : 55442-00-7



〈MW : 400.47 ($C_{26}H_{24}O_4$)〉

(*S*)-2,2'-Binaphthyl-17-crwon-5

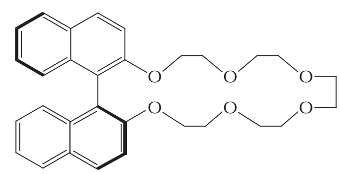
Cat. #020-14711 1 g
RT, Solid (nearly pale yellow, powder)
CAS : 55442-01-8



〈MW : 444.52 ($C_{28}H_{28}O_5$)〉

(*S*)-2,2'-Binaphthyl-20-crwon-6

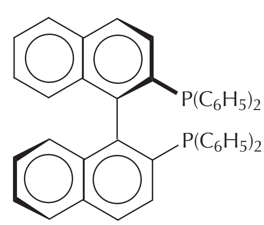
Cat. #024-14731 1 g
RT, Solid (colorless, viscous mass)
CAS : 41024-92-4



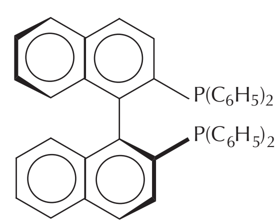
〈MW : 488.57 ($C_{30}H_{32}O_6$)〉

B. Ligands for Catalytic Asymmetric Synthesis: BINAP

BINAP is one of the most frequently used chiral phosphine compounds, which is used for acymmetric synthesis using various kinds of metal catalysts.



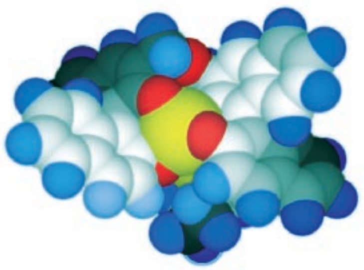
(*R*)-BINAP



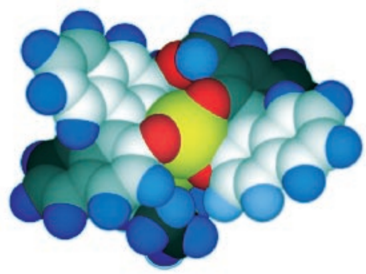
(*S*)-BINAP

Wako Catalog No.	Product	Package Size	MW	CAS	Assay	Appearance	Storage
029-14301 025-14303 027-14302	**(*R*)-(+)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl** [(*R*)-(+)-BINAP]	1 g 5 g 25 g	622.67 ($C_{44}H_{32}P_2$)	76189-55-4	97.0+% (HPLC)	Solid	RT
026-14311 022-14313 024-14312	**(*S*)-(-)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl** [(*S*)-(-)-BINAP]	1 g 5 g 25 g	622.67 ($C_{44}H_{32}P_2$)	76189-56-5		Solid	RT

C. Ligands for Asymmetric Two-Center Catalysis : linked-BINOL

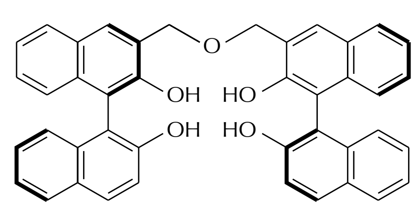


(*R*,*R*)-Ga-Li-linked-BINOL



(*S*,*S*)-Ga-Li-linked-BINOL

The molecule models of Ga-Li-linked BINOL by X-ray crystal structure analysis

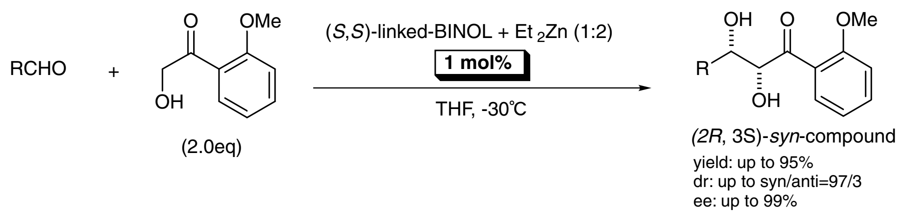


(*S*,*S*)-linked-BINOL

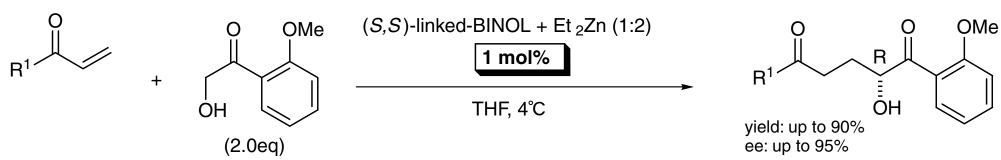
[Features]

Ligands for direct catalytic asymmetric aldol reaction with unmodified ketones and for the catalytic asymmetric Michael reaction

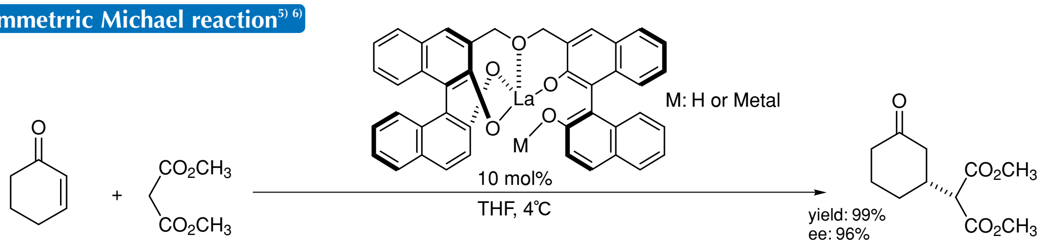
[Reaction 1] **Asymmetrric aldol reaction**[1) 2) 4)]



[Reaction 2] **Asymmetrric Michael reaction**[3) 4)]



[Reaction 3] **Asymmetrric Michael reaction**[5) 6)]



[References]

1) N. Yoshikawa, N. Kumagai, S. Matsunaga, G. Moll, T. Ohshima, T. Suzuki, M. Shibasaki: *J. Am. Chem. Soc.*, **123**, 2466 (2001).
2) N. Kumagai, S. Matsunaga, N. Yoshikawa, T. Ohshima, M. Shibasaki: *Org. Lett.*, **3**, 1539 (2001).
3) N. Kumagai, S. Matsunaga, M. Shibasaki: *Org. Lett.*, **3**, 4251 (2001).
4) M. Shibasaki, M. Kanai, K. Funabashi: *Chem. Commun.*, 1989 (2002).
5) Y. S. Kim, S. Matsunaga, J. Das, A. Sekine, T. Ohshima, M. Shibasaki: *J. Am. Chem. Soc.*, **122**, 6506 (2000).
6) S. Matsunaga, T. Ohshima, M. Shibasaki: *Terathedron Lett.*, **41**, 8473 (2000).

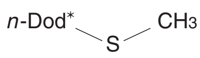
Wako Catalog No.	Product	CAS No.	MW	Pkg. Size	Appearance	Storage
155-02421	**3,3''-[Oxybis(methylene)] bis-(1*R*,1''*R*)-1,1'-bi-2-naphthol** [(*R*,*R*)-linked-BINOL], 90.0+ % (HPLC)	265116-85-6	614.68 ($C_{42}H_{30}O_5$)	200 mg	Solid	RT
152-02431	**3,3''-[Oxybis(methylene)] bis-(1*S*,1''*S*)-1,1'-bi-2-naphthol** [(*S*,*S*)-linked-BINOL], 95.0+ % (HPLC)	336800-79-4	614.68 ($C_{42}H_{30}O_5$)	200 mg		

A. Sulfur Compounds with Less Odor

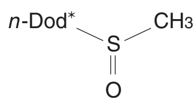
These sulfur compounds with less odor are made by replacing one of alkyl chains bound to a sulfur atom by a dodecyl radical. Each has less volatility and smells less disagreeable.

Recently, sulfur compounds with less odor have been investigated[1) 2)], and Sulfide ① and Sulfoxide ② are each applicable to alcohol oxidation reaction, typified by Corey-Kim oxidation and Swern oxidation. Especially in Corey-Kim oxidation, the reaction progresses even in solvents that are easy to treat. Moreover, Sulfide ① can be applied to dealkylation of ethers and esters. Sulfonium salt ③ can also be used as a methylation agent for micell formation[4) 5)] in addition to synthesis of oxirane[3)].

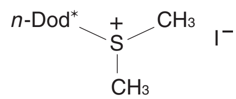
Thiol ④ and ⑤ that introduced the trimethylsilyl (TMS) group, which readily induces functional group conversion, to the benzene ring will be released soon[6)].



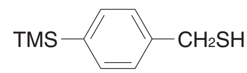
①



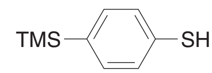
②



③



④

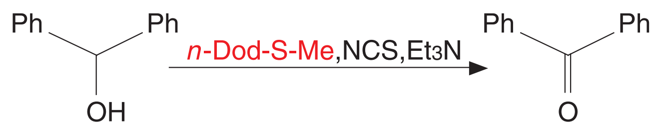


⑤

*: n-Dod=$CH_3(CH_2)_{11}$–

[Example reactions]

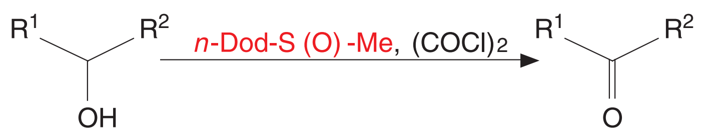
Corey-Kim Oxidation



yield:91～99%

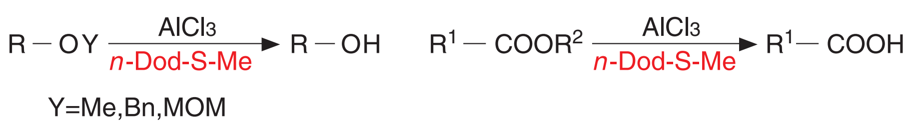
Solvent	Yield (%)
CH_2C_2	96
Toluene	98
AcOEt	97
THF	99
CH_3CN	93
Acetone	100

Swern Oxidation

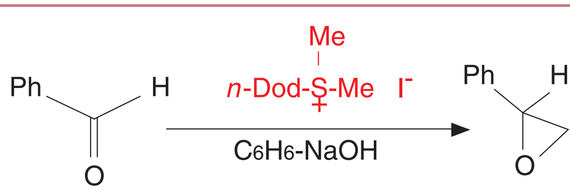


yield:74～95%

Dealkylation



Oxirane synthesis



yield:81%

Wako Catalog No	Description	Package Size
040-28581	Dodecyl Methyl Sulfide	10g
047-28591	Dodecyl Methyl Sulfoxide	10g
040-28601	Dodecyldimethylsulfonium Iodide	10g
209-15961	*p*-(Trimethylsilyl)benzenethiol	1g
205-15963		5g
206-15971	*p*-(Trimethylsilyl)phenylmethanethiol	10g

[References]

1) K. Nishide, S. Ohsugi, H. Shiraki, H. Tamakita, M. Node: *Org. Lett.*, **3**, 3121 (2001).
2) M. Node, K. Kumar, K. Nishide, S. Ohsugi, T. Miyamoto: *Tetrahedron Lett.*, **42**, 9207 (2001).
3) Y. Yano, T. Okonogi, M. Sunaga, W. Tagaki: *J. Chem. Soc., Chem. Commun.*, 527 (1973)
4) K. Yamauchi, Y. Hisanaga, M. Kinoshita: *J. Am. Chem. Soc.*, **105**, 538 (1983),
5) K. Yamauchi, Y. Hisanaga, M. Kinoshita:*J. Chem. Soc. Perkin Trans.*, **1**, 1941 (1983).
6) K. Nishide, T. Miyamoto, K. Kumar, S. Ohsugi, M. Node: *Tetrahedron Lett.*, **43**, 8569 (2002).

Others

B. Condensing agent

Triazine-based Condensing Reagent

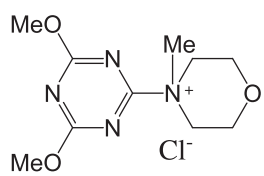
DMT-MM

[4-(4,6-Dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium Chloride n-Hydrate]

The most common technique to form amide and ester bonds is dehydrocondensation accompanied by activation of carboxylic acids. Carbodiimides (WSC, DCC) are widely used as a condensing reagent from reactive and economical standpoints. There are, however, problems that need to be addressed, which are induction of allergy and necessity of removal of byproducts.

Wako introduces a new triazine-based condensing reagent, DMT-MM. DMT-MM can be used under the same reaction condition as for widely used condensing reagents, WSC and DCC. It also can be used in water, protic solvents, hydrated organic solvents and water-organic system. The solvents are not required to dehydrate.

[Structure]



4-(4,6-Dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium Chloride n-Hydrate

[CAS No. 3945-69-5]

[Reaction example]

Amidation

$$\underset{\mathbf{1}}{Ph(CH_2)_2COOH} + \underset{\mathbf{2}}{Ph(CH_2)_2NH_2} \xrightarrow[\text{rt, 3Hr}]{\text{Condensing Agent}} \underset{\mathbf{3}}{Ph(CH_2)_2CONH(CH_2)_2Ph} + Ph(CH_2)_2COOR$$

Run	Cndensing agent	Solvent	Amid (3)[a]	Ester[a] (%)	Amide / Ester
1	DMT-MM	MeOH	98	1.0 (R=Me)	98
2	DMT-MM	EtOH	99	0.7 (R=Et)	141
3	DMT-MM	*i*-PrOH	96	0.7 (R=*i*-Pr)	137
4	DCC	MeOH	27[b]	7.0 (R=Me)	4
5	EDC	MeOH	53	16.0 (R=Me)	3.3

a : Isolated yield
b : *N*-Acylurea was isolated in 21%

Wako Catalog No	Description	Package Size
329-53751	4-(4,6-Dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium Chloride n-Hydrate	5g
327-53752		25g

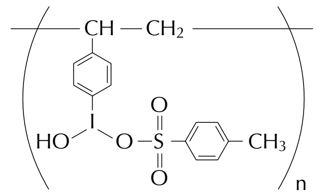
[References]

M. Kunishima, C. Kawachi, K. Hioki, K. Terao, S. Tani : *Tetrahedron*, **57**, 1551 (2001).

C. Tosyloxylation Reaction

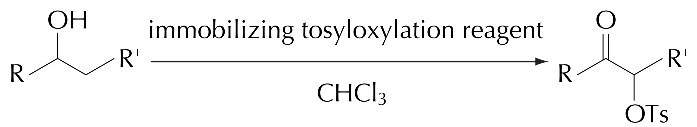
Poly [*p*-(hydroxy) (tosyloxy) iodostyrene]

Cat. #163-20461 500 mg
−20℃, Solid
Molecular Formula : -[$C_{15}H_{15}IO_4S$]$_n$-



[Features]
1. For oxidative α-tosyloxylation of alcohols, α-tosyloxylation of ketones and oxidative α-tosyloxylation of alkynes
2. Readily isolated from the reaction mixture by filtration

[Reaction]



Yield : up to 98%

Oxidative α-tosyloxylation of alcohols

[References]
1) H. Togo and K. Sakuratani: *Synlett*, 1966 (2002)
2) S. Abe, K. Sakuratani and H. Togo: *Synlett*, 22 (2001)
3) S. Abe, K. Sakuratani and H. Togo: *J. Org*. Chem., **66**, 6174 (2001)

D. CFC Alternatives HCFC-225

HCFC-225 is a highly safe fluorinated solvent with no flash point. Since it also has excellent features such as low surface tension, low viscosity and selective solubility, it is widely used not only as a detergent, but also as a functional solvent in various fields of industry. HCFC-225 is highly appreciated internationally as the alternative to CFC-113 and is listed as an acceptable substance in the SNAP (Significant New Alternative Policy) Program under the US Clean Air Act.

[Features]
1. Physical properties is similar to CFC-113
2. Cleaning performance is similar to or better than CFC-113
3. Existing cleaning equipment can be used with no modification
4. Non-flammable
5. Compatible with most plastics and metals
6. Low boiling point and short drying time
7. Excellent permeability due to low surface tension
8. High chemical and heat stability
9. Easy solution management
10. Recyclable by distillation

Description		HCFC-225
Chemical formula		$CF_3CF_2CHCl_2$ / $CClF_2CF_2CHClF$
Boiling point	℃	54
Freezing point	℃	-131
Viscosity	mPa・S	0.59
Surface tension	mN/m	16.2
Flash point	℃	nil

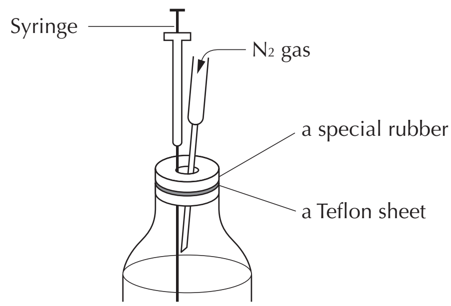
Reference values H_2O (25℃) : Viscosity 1.0mPa・S、Surface tension 72.0mN/m

Wako Catalog No	Description	Package Size
321-39232	HCFC-225 (mixture of $CF_3CF_2CHCl_2$ and $CClF_2CF_2CHClF$)	25g
325-39235		500g

E. Dehydrated Solvents

Solvent for organic synthesis, containing its water content to its minimum. Can be used as a solvent for various organic synthesis reactions where water should be avoided. Each 100mL-and 500 mL-package has a special cap through which a syringe needle can be inserted. As shown in the figure, the solvent is collected while blowing nitrogen gas into a package.

100mL-and 500mL-package



Product Name	Water content	Package Size			Class	UN	Storage
		100 mL	500 mL	3 L			
Acetone, Dehydrated (99.5+ %)	max. 50 ppm	010-15533	016-15535	014-15531	3	1090	Protect from light
Acetonitrile, Dehydrated (99.0+ %)	max. 50 ppm	017-15543	013-15545	011-15541	3	1648	RT
Benzene, Dehydrated (99.5+ %)	max. 30 ppm	022-12853	028-12855	026-12851	3	1114	RT
1-Butanol, Dehydrated (99.0+ %)	max. 50 ppm	-	020-13035	028-13031	3	1120	RT
2-Butanone, Dehydrated (99.0+ %)	max. 50 ppm	-	027-13045	025-13041	3	1193	Protect from light
Butyl Acetate, Dehydrated (99.0+ %)	max. 50 ppm	027-13263	023-13265	-	3	1123	RT
Chloroform, Dehydrated, containing 0.3 ~ 1.0 % of Ethanol (99.0+ %)	max. 30 ppm	035-16283	031-16285	039-16281	6.1	1888	RT
Chloroform, Dehydrated, containing 150 ppm of Amylene (99.0+ %)	max. 30 ppm	032-16813	038-16815	036-16811	6.1	1888	RT
Cyclohexane, Dehydrated (99.5+ %)	max. 30 ppm	-	036-16595	034-16591	3	1145	RT
Dichloromethane, Dehydrated (99.0+ %)	max. 30 ppm	048-25503	044-25505	042-25501	6.1	1593	Protect from light
Diethyl Ether, Dehydrated (99.5+ %)	max. 50 ppm	-	041-25495	-	3	1155	Protect from light
***N,N*-Dimethylacetamide, Dehydrated** (98.0+ %)	max. 50 ppm	-	042-25285	040-25281	-	-	RT
***N,N*-Dimethylformamide, Dehydrated** (99.5+ %)	max. 50 ppm	041-25473	047-25475	045-25471	3	2265	Protect from light
Dimethyl Sulfoxide, Dehydrated (99.0+ %)	max. 50 ppm	046-26023	042-26025	-	-	-	RT
1,4-Dioxane, Dehydrated (99.0+ %)	max. 50 ppm	-	044-25485	042-25481	3	1165	RT
Ethanol, Dehydrated (99.5+ %)	max. 50 ppm	055-06133	051-06135	059-06131	3	1170	RT
Ethyl Acetate, Dehydrated (99.5+ %)	max. 50 ppm	050-06183	056-06185	054-06181	3	1173	RT
Ethylene Glycol, Dehydrated (99.5+ %)	max. 50 ppm	053-06313	059-06315	-	-	-	RT
Heptane, Dehydrated (99.0+ %)	max. 30 ppm	089-07273	085-07275	-	3	1206	RT
Hexane, Dehydrated (96.0+ %)	max. 30 ppm	089-07033	085-07035	083-07031	3	1208	RT
Methanol, Dehydrated (99.8+ %)	max. 50 ppm	136-12383	132-12385	130-12381	3 & 6.1	1230	RT
4-Methyl-2-pentanone, Dehydratede	max. 50 ppm	131-12713	137-12715	-	3	1245	Protect from light
1-Methyl-2-pyrrolidone, Dehydrated (97.0+ %)	max. 50 ppm	138-12723	134-12725	-	-	-	RT
1-Propanol, Dehydrated (99.5+ %)	max. 50 ppm	-	166-18305	164-18301	3	1274	RT
2-Propanol, Dehydrated, containing Iso-propanol (99.5+ %)	max. 50 ppm	165-17993	161-17995	169-17991	3	1219	RT
Pyridine, Dehydrated (99.5+ %)	max. 50 ppm	161-18453	167-18455	165-18451	3	1282	RT
Tetrahydrofuran, Dehydrated, containing 0.03% BHT (99.5+ %)	max. 50 ppm	206-13433	202-13435	200-13431	3	2056	Protect from light
Tetrahydrofuran, Dehydrated, containing no stabilizer (99.5+ %)	max. 50 ppm	207-13963	203-13965	201-13961	3	2056	Protect from light
Toluene, Dehydrated (99.5+ %)	max. 30 ppm	203-13443	209-13445	207-13441	3	1294	RT
Xylene, Dehydrated (80+ %) (mixture of *o*-, *m*- and *p*-Xylene)	max. 30 ppm	-	242-00685	240-00681	3	1307	RT

Chromato Sheet

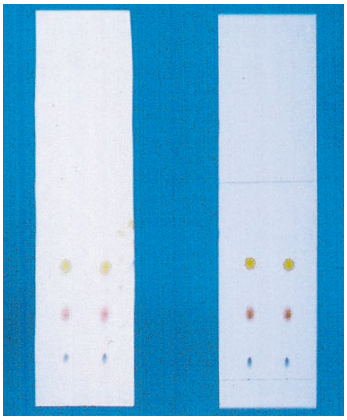
Cat. #036-17151   25 sheets

RT

Chromato sheet is an environment-friendly product, making full use of advantages of "paper." It is light, easy to use and disposable with no damage to environment. Unlike a conventional thin-layer chromatoplate (TLC plate), this is made from paper fiber which is coated with silica gel and fluorescent F254. This product provides usability of paper and separabiltiy of silica gel (adsorption/partition mode).

[Features]

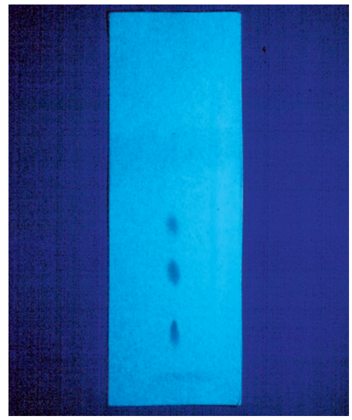
1. High resolution & reproducibility
2. Clipping, Wiring and Filing as a paper
3. Meet no-detached silica gel powder
4. Applicable to blotting
5. Applicable to fluorometric detection



Left : Chromato Sheet
Right : Silicagel 70F254 Plate-wako
Sample : Wakogel B-Tester (#231-00051)
Developing solvent : Chloroform

[Specification]

Size : 20 x 20 cm
Weight : Approximately 7 g/sheet
Thickness : 0.3 mm
Silica gel : Wakogel C-500HG containing F254 fluorescent Indicator.

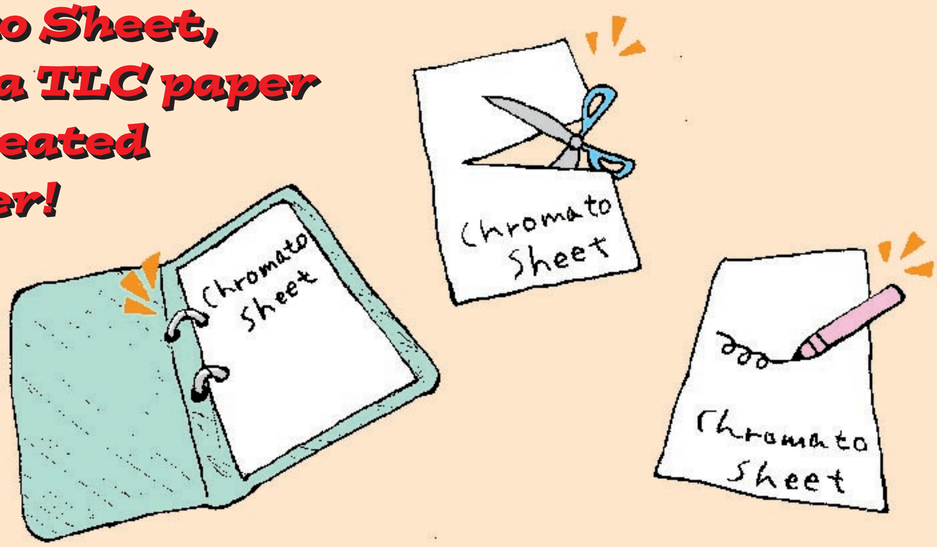


Detection at 254 nm
Sample : Brucine, oxypropyltheophylline and caffein
Developing solvent : Chloroform(9) + Methanol(1)

[Limitation]

Inapplicable to use color-producing reagents containing strong acids and to treat carbonization by heating at a high temperature

Chromato Sheet, which is a TLC paper can be treated as a paper!



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