

# Metathesis Application Guide

Grubbs Catalyst® Technology  
for Chemical Synthesis



The life science  
business of Merck  
KGaA, Darmstadt,  
Germany operates as  
MilliporeSigma in the  
U.S. and Canada.

**Sigma-Aldrich®**  
Lab & Production Materials

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# Expertise in Metathesis Catalysts

## Empowering Breakthrough Synthesis

We are committed to helping you reach new frontiers through an ever-expanding, always innovative portfolio of products. In line with this goal, we have partnered with Umicore PMC to bring you an outstanding range of olefin metathesis catalysts for chemical synthesis.

Our exclusive collaboration not only gives you direct access to Umicore's Grubbs Catalyst® technology, but also their development expertise. You'll enjoy rapid, reliable supply of milligram to multi-kilogram volumes, at the best value, with the Umicore license rights included. Moreover, you'll benefit from dedicated technical guidance, supported by Nobel Laureate Robert Grubbs. Discover an unparalleled portfolio of metathesis catalysts for your breakthrough synthesis ideas.

## Umicore Precious Metals Chemistry

Umicore Precious Metals Chemistry (PMC) is part of the Umicore group, a global materials and technology company. With over 50 years of experience working with customers across the world, Umicore PMC has developed innovative metals and materials technology to help solve the most challenging chemistry problems. The company's extensive portfolio of chemical technologies includes the Grubbs metathesis, cross coupling and hydrogenation catalysts.

Umicore PMC operates in many key markets that are vital to developing solutions to real-world problems. This includes: pharmaceutical development, fine chemicals, automotive and electronics. Within these industries, the company offers many competitive technologies that deliver ground-breaking innovations. Taking a collaborative approach to its business, Umicore PMC works closely with customers in multiple aspects of R&D, process development and industrial manufacturing to create novel, cost-effective synthesis solutions.

In addition to the company's chemical and engineering expertise, sustainability lies at the heart of the Umicore PMC business and the wider Umicore group. Sustainability is materialized through Umicore PMC's drive towards developing efficient methods of recycling precious metals, and discovering new, more long-term sustainable chemical reactions.

## The Best of Metathesis Catalysts

First established in the 1960s, alkene metathesis is considered the best method to synthesize long complex alkenes through the use of highly efficient and selective

catalytic reactions. Employing a ruthenium metal complex, metathesis enables the simple synthesis of carbon-carbon double bonds. These reactions provide efficient routes to product synthesis by ensuring minimal waste due to the formation of less toxic by-products, high activity and high stereoselectivity.

Following decades of research and development in the field of metathesis, Umicore PMC provides a comprehensive portfolio of catalysts. In early 2018, the company expanded its offerings to include the world-class Grubbs Catalyst® intellectual property, following its acquisition of Materia's proprietary metathesis technologies. These catalysts, developed by Nobel Laureate Prof. Robert H. Grubbs and his team, deliver best-in-class performance, enabling robust alkene conversions.

Umicore PMC now offers secured access to the broadest catalyst portfolio for metathesis. This is in addition to the strong global operating and industrial scale manufacturing expertise that it has established over its decades of experience in the industry. The company's range of services are tailored to meet customer's specific metathesis needs. Umicore PMC experts can help support development processes; from scaling a reaction or devising new reaction routes, to screening the appropriate catalyst to maximize product formation.

### About this Guide

Along with the experts at Umicore PMC, we have created this practical guide to help you apply olefin metathesis in your own synthetic routes. It starts by discussing general reaction parameters and practical considerations for running routine olefin metathesis reactions. It then covers some more challenging metathesis reactions with examples from academic, pharmaceutical, and specialty chemical laboratories, which illustrate some elegant solutions that have been developed. Finally, it provides a quick guide to selecting the appropriate metathesis catalyst for your specific synthesis.

Access an unparalleled portfolio of metathesis catalysts for your breakthrough synthesis:

[SigmaAldrich.com/Grubbs](https://www.sigmaaldrich.com/Grubbs)

# Metathesis: Key Features

## **Empowering Breakthrough Synthesis**

Due to its versatility, metathesis can be used to synthesize a range of useful products across multiple markets. The key features of metathesis are described below.

## **Functional Group Tolerance**

Modern active pharmaceutical ingredients are among the most complex molecules synthesized at commercial scale. Ruthenium-based metathesis catalysts succeed in forming key bonds in even the most challenging substrates, such as the macrocyclic peptide core of HCV NS3 protease inhibitors like ciluprevir, simeprevir, and related compounds used to treat hepatitis C infections.

## **Stereoselectivity**

Recent advances in ruthenium-based metathesis catalysts offer opportunities for stereoselective metathesis reactions. In particular, Z-selective and stereoretentive catalysts offer new routes for chemists to control the E:Z ratio of newly formed alkenes. While this feature is potentially useful in almost any application, it is particularly advantageous to the production of insect pheromones, which must be prepared in the correct ratio of olefin isomers to be effective as natural deterrents for crops.

## **Activity and Catalyst Longevity**

For chemical applications driven by aggressive cost targets, ruthenium-based metathesis catalysts have demonstrated unparalleled activity under rugged conditions. Renewable feedstocks such as soybean oil can be converted into specialty chemical products with turnover numbers in the hundreds of thousands. Thus, even in high-volume, low-margin products, metathesis can deliver significant cost advantages.

# General Reaction Procedures

Metathesis reactions are versatile reactions that can be performed in most situations without the need for extensive optimization. But for more difficult synthetic situations, Umicore PMC's team of industrial experts will tailor each reaction to the specific requirements for the project, ensuring precise and selective product formation. There are many factors that must be considered when devising any reaction, including those from catalyst loadings, reaction conditions and the functionality of the product.

## Metathesis Reaction Types

There are three main classes of metathesis reactions, two of which are used regularly for small molecule organic synthesis. Ring-closing metathesis is an intramolecular reaction of an acyclic diene to form a ring (Fig. 1), while cross metathesis brings two olefins together in an intermolecular reaction to give an olefin product bearing substituents from each of the starting olefins (Fig. 2).

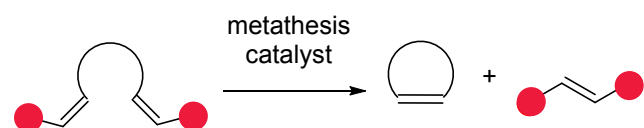


Figure 1. Ring-closing metathesis (RCM)

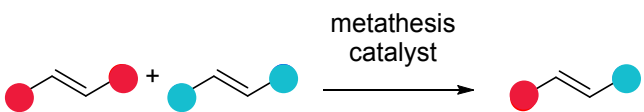


Figure 2. Cross metathesis (CM)

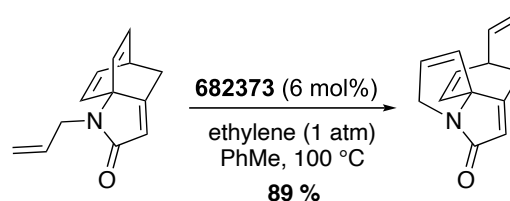
## General Reaction Set-Up

1. Deoxygenated solvents and reaction mixtures are recommended for optimal results. If necessary, degas the solvent before use.
2. In a dry, inert reaction vessel with a stir bar, dissolve your substrate(s) in the solvent of choice.
3. Weighing the catalyst open to the air is fine. The reaction vessel should be pumped and purged with inert gas before dissolution of catalyst, whether the solid catalyst will be used directly or added as a solution. The use of degassed solvents is highly recommended, and the solvent should be degassed before it is added to the catalyst.
4. Heat the reaction to the desired temperature and monitor until complete.

## Optimizing the Catalyst

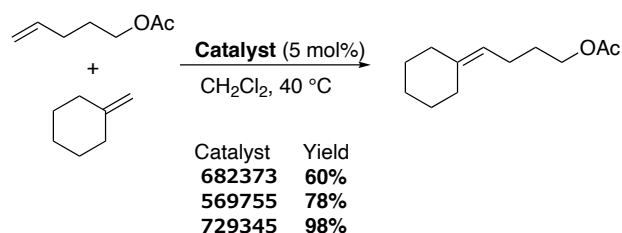
For general metathesis reactions, Umicore Grubbs Catalyst<sup>®</sup> M202 and M204 as well as the Umicore Hoveyda-Grubbs Catalyst<sup>®</sup> M720 and M730 are recommended. Hoveyda-type catalysts initiate at room temperature and are highly stable, enabling simple storage and handling.

In metathesis reactions involving sterically hindered alkenes, it may be necessary to use a more specialized catalyst. For instance, Umicore Hoveyda-Grubbs Catalyst<sup>®</sup> M721, M722 or M731 can be used to perform sterically hindered ring rearrangement reactions owing to the decreased steric bulk of the protruding ligands.



Ref.: Bioorg. Med. Chem. 2013, 21, 5707.

Alternatively, if the substrate's substituents are bulky, substituted alkenes, the Umicore Hoveyda-Grubbs Catalyst<sup>®</sup> M722 could result in a higher yield.



Ref.: Org. Lett. 2008, 10, 441.

As long as reaction conditions are chosen carefully, high loadings of catalyst are not necessary. In some cases, using more catalyst may lead to unwanted side reactions. Even for challenging reactions, loadings less than 1 mol% can be effective given the careful choice of other reaction parameters.

## Choosing the Right Conditions

Selecting the right operating conditions for your industrial reaction relies on careful optimization. Finding the right temperature is vital for catalyst initiation, with low temperatures being advantageous from an environmental perspective. Umicore catalysts typically initiate at low temperatures, between room temperature and 40°C.

Furthermore, the exact concentrations of reagents vary according to the specific metathesis reaction: cross metathesis requires concentrated solutions, macrocyclizations require dilute solutions, and other ring-closing metathesis reactions require intermediate concentrations.

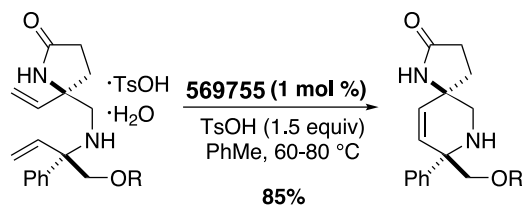
Likewise, choosing the appropriate solvent to mediate metathesis reactions relies on understanding the various properties of the solvents. Preferred solvents include non-polar, hydrocarbon-based solvents, chlorinated solvents and peroxide-resistant ethers, due to their weak binding affinity to the catalyst complex.

Preferred Solvents	Suitable in Certain Conditions	Not Recommended
Toluene, xylenes, mesitylene	MeOH, EtOH, nBuOH	DMSO, DMF, NMP
Heptanes, hexanes	THF, ether	MeCN
DCM, DCE, chlorobenzene	Water (neutral/acidic)	Pyridine and other amines
EtOAc, iPrOAc		Water (basic)
TBME, Me-THF		

Finally, it is also vital to ensure that the catalytic reaction is devoid of catalyst poisons. Peroxides oxidize the metal-carbene bond, rendering the catalyst inactive. Ethene should also be removed as its presence could result in catalyst decomposition. In addition, all of the Umicore Hoveyda-Grubbs Catalysts® are air and moisture stable as solids, but in solution are vulnerable to oxygen. Therefore, reactions need to be under argon or nitrogen atmosphere to ensure the exclusion of oxygen.

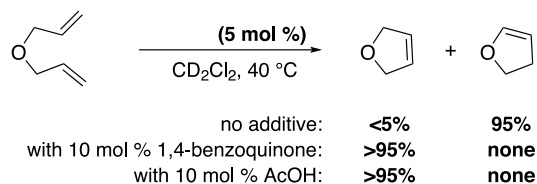
## Optimizing the Reaction Procedure

Beyond conditions, there are multiple aspects of the synthesis procedure that must be considered for successful completion of any reaction. Strongly coordinating functional groups must be masked so as to not disrupt the catalyst activity; the concentration of the various substrates must be optimized to prevent substrate polymerization, but encourage the desired format of metathesis.



Ref.: Wu, George G. et al (Schering-Plough, USA)  
WO 2010028232, Mar 11, 2010.

Furthermore, to prevent unwanted side-effects of metathesis reactions, such as the isomerization of alkenes, it is sometimes necessary to use additives. For instance, mild acids such as acetic acid can be added to reactions to prevent hydride formation, as illustrated in the following reaction scheme:



Ref.: J. Am. Chem. Soc. 2003, 125, 2546; J. Am. Chem. Soc. 2005, 127, 17160.

Metathesis reactions that bring together two terminal alkenes produce ethene as a by-product. Although ethene is a gas, it is soluble in organic solvents and can remain in the reaction mixture. Ensuring that ethene or any other gaseous by-product is efficiently removed will drive the reaction equilibrium toward completion. This can be accomplished by bubbling an inert gas through the reaction mixture over the course of the reaction. On scale, this technique is used frequently to help maximize catalyst lifetime.

# Choose Your Metathesis Route

With many versatile reaction options available, selecting the right catalyst also relies on identifying the right metathesis reaction. Be it ring-closing metathesis, which can be used to synthesize complex polycyclic molecules, or cross metathesis, involving the intermolecular reaction of two unconnected alkenes, Umicore PMC offers hands-on expertise to discuss the best routes to your product, ensuring project success.

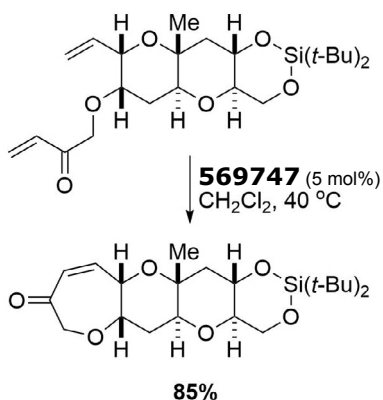
## 1. Ring-Closing Metathesis

Ring-closing metathesis is a common metathesis reaction for any scientist needing to synthesize mid- and macro-sized rings, as well as offering an efficient route to synthesize strained or sterically hindered rings. Proceeding via an intermolecular process, this reaction can be made almost irreversible following appropriate reaction optimization.

### Mid-sized ring-closing metathesis

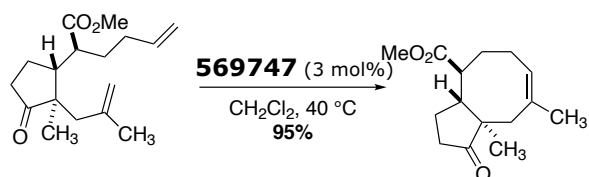
Preferred Catalysts	Optimal Catalyst Loading	Conditions
Umicore Grubbs Catalyst® M202	3-5 mol%	Concentration: (depending on ring size) 1.0 M (5-membered ring); 0.5 M (6-membered ring)
Umicore Grubbs Catalyst® M204		
Umicore Hoveyda-Grubbs Catalyst® M720		
Umicore Hoveyda-Grubbs Catalyst® M721		
Umicore Hoveyda-Grubbs Catalyst® M730		
		Temperature: 40 – 100°C

Ring-closing metathesis to form an oxepane ring embedded in (-)-gambieric acid with applications in the pharmaceutical industry:



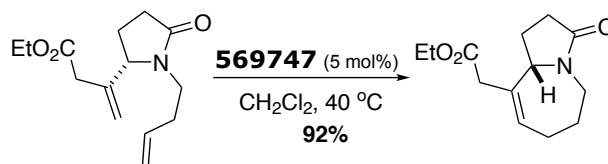
Ref.: Organic Letters 2015, 17, 4694.

Ring-closing metathesis to yield the synthesis of an 8-membered ring structure of serpendione:



Ref.: Tetrahedron Letters 2005, 46, 1149.

Ring-closing metathesis in the synthesis of (-)-stemoamide, a root extract used in Chinese and Japanese folk medicine:

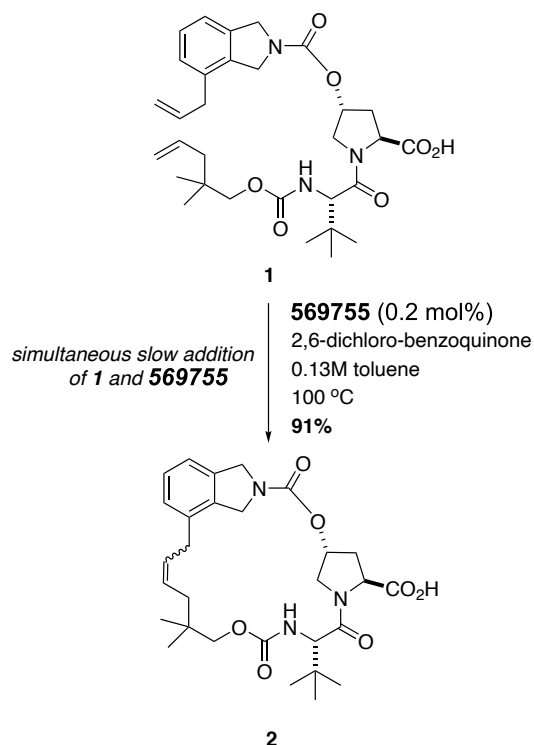


Ref.: Journal of Organic Chemistry 2007, 72, 4246.

### Macrocyclic ring-closing metathesis

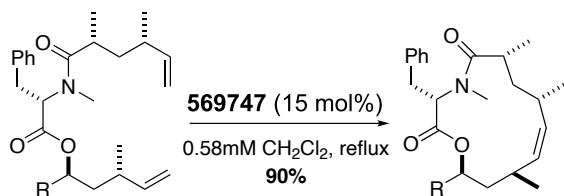
Preferred Catalysts	Optimal Catalyst Loading	Conditions
Umicore Grubbs Catalyst® M202	3-10 mol%	Concentration: (depending on ring size) 1.0 M (5-membered ring); 0.5 M (6-membered ring)
Umicore Grubbs Catalyst® M204		
Umicore Hoveyda-Grubbs Catalyst® M720		
Umicore Hoveyda-Grubbs Catalyst® M731		Temperature: 40 – 100°C

Ring-closing metathesis to form a 20-membered macrocycle used as a protease inhibitor in the pharmaceutical industry:



Ref.: Journal of Organic Chemistry 2012, 77, 3820.

Formation of a key intermediate in the preparation of the cytotoxic marine natural product (-)-spongidepsin:

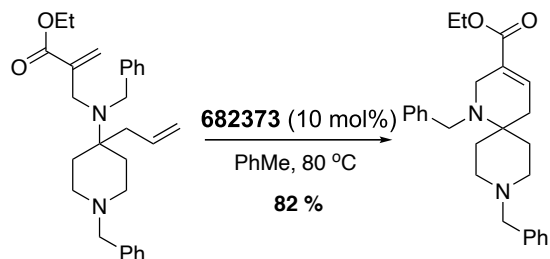


Ref.: Organic Letters 2010, 12, 4392.

### Sterically demanding ring-closing metathesis

Preferred Catalysts	Optimal Catalyst Loading	Conditions
Umicore Hoveyda-Grubbs Catalyst® M721	3-10 mol%	Concentration: (depending on ring size) 1.0 M (5-membered ring); 0.5 M (6-membered ring)  Temperature: 40 – 100°C

Formation of a trisubstituted alkene scaffold used for SAR exploration:



Ref.: Bioorganic Medicinal Chemistry 2013, 21, 5707

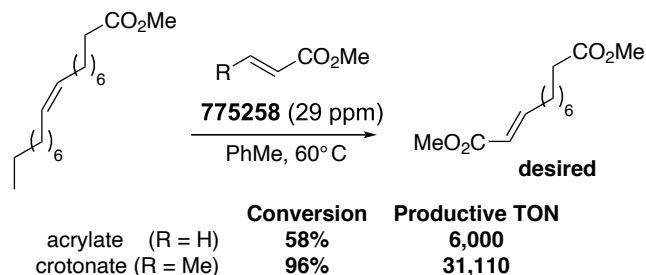
## 2. Cross Metathesis

Bringing together two unconnected alkenes in an intermolecular reaction, cross metathesis is an extremely useful reaction that can result in the efficient synthesis of complex and long carbon-carbon chains.

### Cross metathesis of electron-deficient alkenes

Preferred Catalysts	Optimal Catalyst Loading	Conditions
Umicore Grubbs Catalyst® M202	1-5 mol%	Concentration: 1.0 M or greater  Temperature: 40 – 60°C
Umicore Grubbs Catalyst® M204		
Umicore Hoveyda-Grubbs Catalyst® M720		
Umicore Hoveyda-Grubbs Catalyst® M730		
Umicore Hoveyda-Grubbs Catalyst® M731		

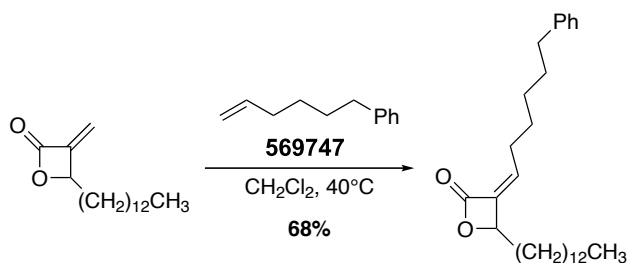
Biscarbocyclic acid formation used as precursors to multiple fine chemical products:



Ref.: ChemSusChem 2014, 8, 1143.



Synthesis of  $\beta$ -lactone structures bearing a variety of alkyl chains at the 3-position:

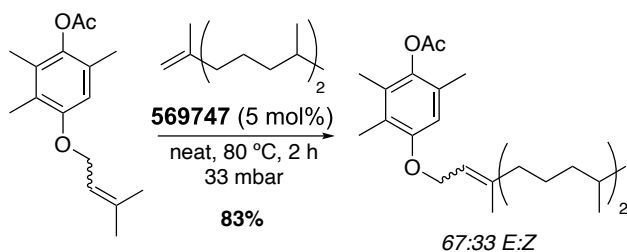


Ref.: Bioorganic & Medicinal Chemistry Letters 2015, 25, 317.

### Synthesis of trisubstituted linear alkenes

Preferred Catalysts	Optimal Catalyst Loading	Conditions
Umicore Grubbs Catalyst® M202	1-5 mol%	Concentration: 1.0 M or greater
Umicore Grubbs Catalyst® M204		Temperature: 40 – 60°C
Umicore Hoveyda-Grubbs Catalyst® M720		
Umicore Hoveyda-Grubbs Catalyst® M722		
Umicore Hoveyda-Grubbs Catalyst® M730		

The preparation of vitamin E intermediates by cross metathesis of trisubstituted and disubstituted alkenes:



Ref.: Helvetica Chimica Acta 2006, 89, 797.

# Metathesis Quick Guide

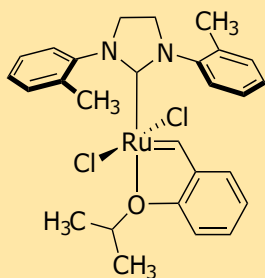
- **Hoveyda-Grubbs Catalyst® M721, Umicore**

For ring-closing metathesis of sterically challenging substrates

**Molecular Weight:** 570.52

**CAS:** 927429-61-6

**Catalog No.** 682373



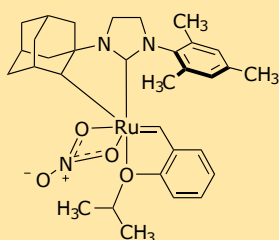
- **Hoveyda-Grubbs Catalyst® M2001, Umicore**

For Z-selective reactions

**Molecular Weight:** 632.76

**CAS:** 1352916-84-7

**Catalog No.** 771082



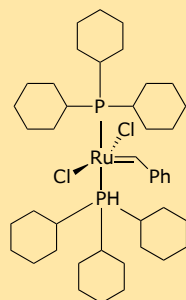
- **Grubbs Catalyst® M102, Umicore**

Grubbs Catalyst® 1st Generation: The original catalyst that started it all

**Molecular Weight:** 822.95

**CAS:** 172222-30-9

**Catalog No.** 579726



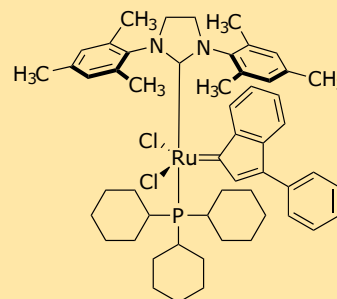
- **Grubbs Catalyst® M202, Umicore**

For alkene, cross, enyne, ring arrangement, and ring closing metathesis reactions

**Molecular Weight:** 949.09

**CAS:** 536724-67-1

**Catalog No.** 775258



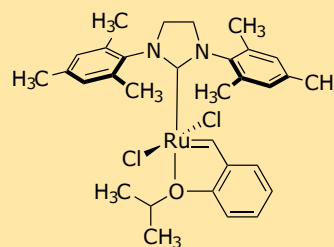
- **Hoveyda-Grubbs Catalyst® M720, Umicore**

Hoveyda-Grubbs Catalyst® 2nd Generation: The go-to for any metathesis reaction

**Molecular Weight:** 626.62

**CAS:** 301224-40-8

**Catalog No.** 569755



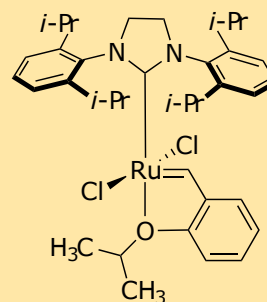
- **Hoveyda-Grubbs Catalyst® M722, Umicore**

For cross metathesis of sterically challenging substrates

**Molecular Weight:** 710.78

**CAS:** 635679-24-2

**Catalog No.** 729345



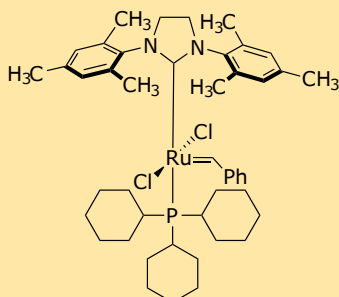
- **Grubbs Catalyst® M204, Umicore**

Grubbs Catalyst® 2nd Generation: The most published and well understood

**Molecular Weight:** 848.97

**CAS:** 246047-72-3

**Catalog No.** 569747



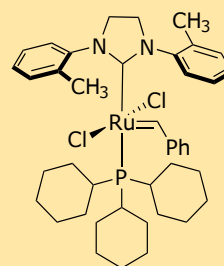
- **Grubbs Catalyst® M205, Umicore**

For preparation of tetrasubstituted olefins via ring closing metathesis

**Molecular Weight:** 729.87

**CAS:** 927429-60-5

**Catalog No.** 682284



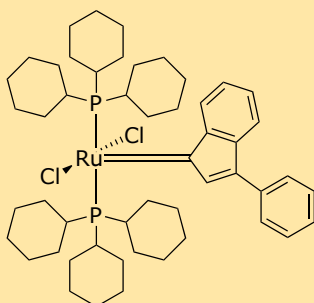
- **Grubbs Catalyst® M101, Umicore**

For ring-closing metathesis, enyne cycloisomerization, nucleophilic additions of acids to alkynes, and diastereoselective double ring-closing metathesis

**Molecular Weight:** 923.07

**CAS:** 250220-36-1

**Catalog No.** 774901



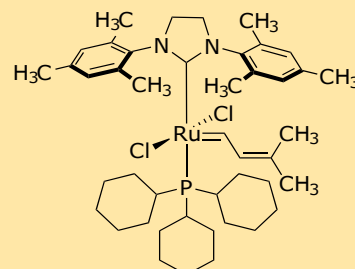
- **Grubbs Catalyst® M207, Umicore**

A general purpose olefin metathesis catalyst similar in reactivity to Grubbs Catalyst® 2nd Generation

**Molecular Weight:** 826.97

**CAS:** 253688-91-4

**Catalog No.** 682365



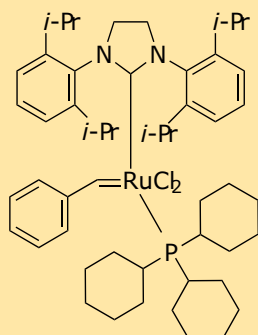
- **Grubbs Catalyst® M206, Umicore**

Bulky NHC ligand results in faster initiation than the Grubbs Catalyst® 2nd Generation

**Molecular Weight:** 933.13

**CAS:** 373640-75-6

**Catalog No.** 729353



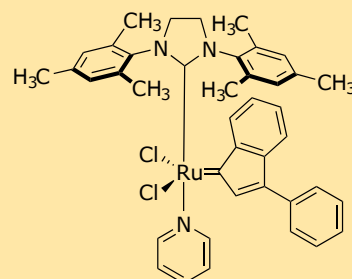
- **Grubbs Catalyst® M310, Umicore**

For cross metathesis of functionalized allylic alcohols, ring-opening metathesis polymerization, and ring-closing metathesis

**Molecular Weight:** 747.76

**CAS:** 1031262-76-6

**Catalog No.** 775282



# Metathesis Quick Guide

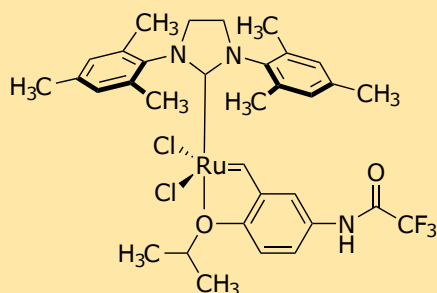
- **Hoveyda-Grubbs Catalyst® M710, Umicore**

For cross metathesis of terpenoids and formation of trisubstituted alkenes via cross metathesis

**Molecular Weight:** 737.64

**CAS:** 1025728-56-6

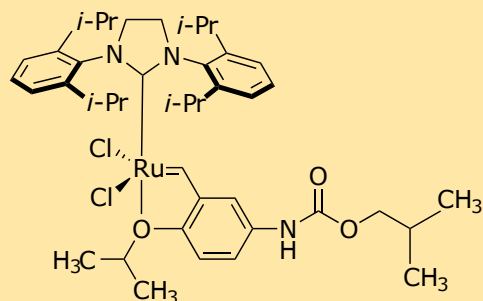
**Catalog No.** 775398



**Molecular Weight:** 825.91

**CAS:** 1212009-05-6

**Catalog No.** 775347



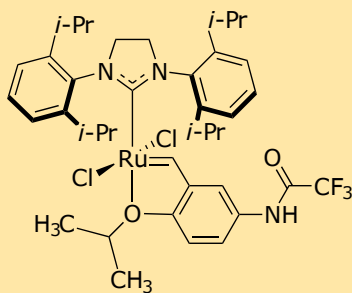
- **Hoveyda-Grubbs Catalyst® M711, Umicore**

For ring-closing metathesis of functionalized  $\alpha,\omega$ -diacids and enynes, cross metathesis with methyl acrylate, and formation of trisubstituted alkenes via cross metathesis

**Molecular Weight:** 821.80

**CAS:** 1212008-99-5

**Catalog No.** 909165



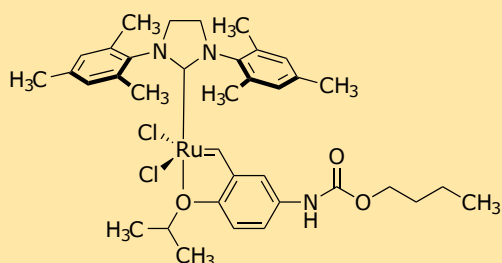
- **Hoveyda-Grubbs Catalyst® M730, Umicore**

For cross metathesis of terpenoids and ring-closing metathesis of functionalized  $\alpha,\omega$ -diacids

**Molecular Weight:** 741.75

**CAS:** 1025728-57-7

**Catalog No.** 775428



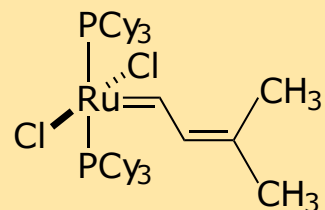
- **Grubbs Catalyst® M103, Umicore**

Precursor to a 2nd Generation Grubbs Catalyst® that is effective for forming trisubstituted olefins

**Molecular Weight:** 800.95

**CAS:** 194659-03-5

**Catalog No.** 908614



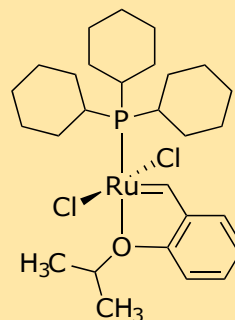
- **Hoveyda-Grubbs Catalyst® M700, Umicore**

For macrocyclization reactions to form disubstituted olefins

**Molecular Weight:** 600.61

**CAS:** 203714-71-0

**Catalog No.** 577944



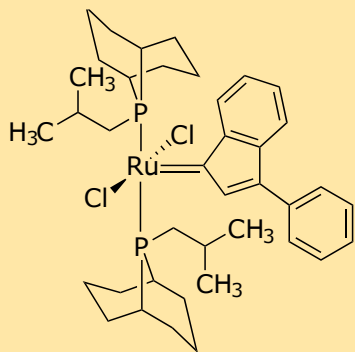
### Grubbs Catalyst® M110, Umicore

For ring-opening metathesis polymerization, ethenolysis of methyl oleate, and self-metathesis of terminal alkenes

**Molecular Weight:** 758.79

**CAS:** 894423-99-5

**Catalog No.** 774928



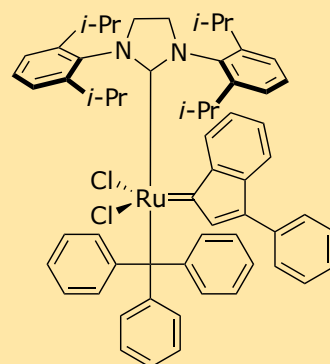
### Grubbs Catalyst® M201, Umicore

For ring-closing enyne metathesis, cross metathesis of terminal alkenes with acrylates, and ring-closing metathesis

**Molecular Weight:** 1015.11

**CAS:** 1307233-23-3

**Catalog No.** 775274



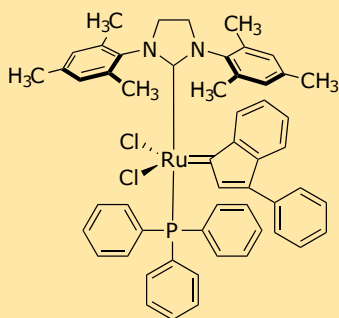
### Grubbs Catalyst® M200, Umicore

For ring-rearrangement metathesis, ring-closing metathesis of enynes, and ring-opening metathesis polymerization

**Molecular Weight:** 930.95

**CAS:** 340810-50-6

**Catalog No.** 775266



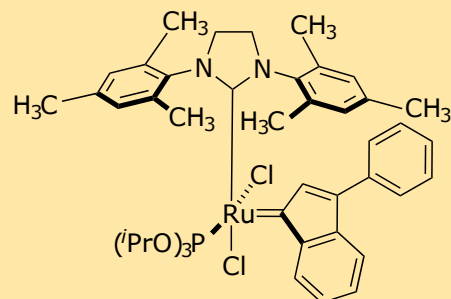
### Grubbs Catalyst® M220, Umicore

For ring-closing metathesis forming trisubstituted C-C double bonds and nitrogen containing heterocycles and cross metathesis of terminal alkenes with acrolein

**Molecular Weight:** 876.91

**CAS:** 1255536-61-8

**Catalog No.** 910430



# Metathesis Quick Guide

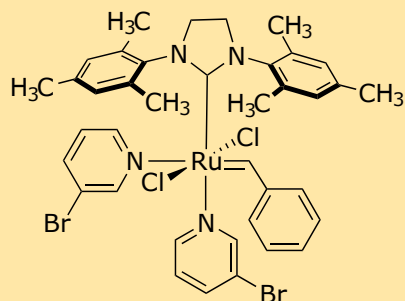
- **Grubbs Catalyst® M300**

For cross metathesis of acrylonitril; for ring closing metathesis and the production of block copolymers by ring opening metathesis polymerisation (ROMP)

**Molecular Weight:** 884.54

**CAS:** 900169-53-1

**Catalog No.** 682330



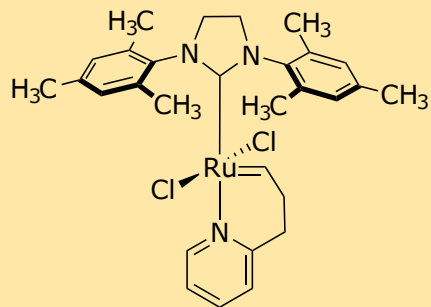
- **Grubbs Catalyst® M360**

For ring opening metathesis polymerisation (ROMP) applications where longer monomer/catalyst resin handling times are desired

**Molecular Weight:** 597.58

**CAS:** 802912-44-3

**Catalog No.** 682381

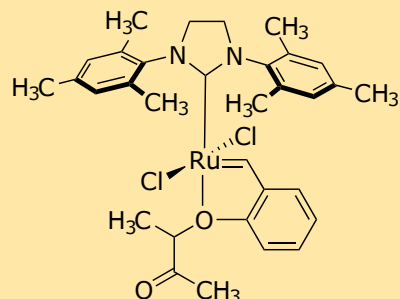


- **Grubbs Catalyst® M510, Umicore**

**Molecular Weight:** 654.63

**CAS:** 1031262-71-1

**Catalog No.** 775312

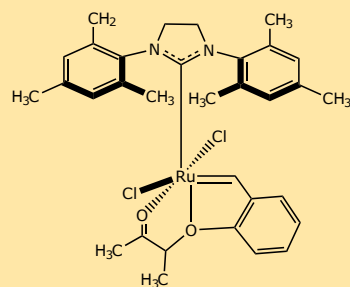


- **Grubbs Catalyst® M520**

**Molecular Weight:** 640.61

**CAS:** 1014701-61-1

**Catalog No.** 775320



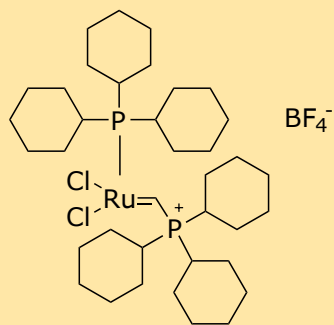
- **Grubbs Catalyst® C833**

Reactivity profile is similar to Grubbs Catalyst® 1st generation but is active at much lower temperatures (below 0 °C)

**Molecular Weight:** 832.66

**CAS:** 1020085-61-3

**Catalog No.** 707961



- **KitAlysis High-Throughput Medium (5, 6, 7) Ring Closing Metathesis Reaction Screening Kit**

Screening kit for ring closing metathesis

**Catalog No.** KITALYSIS-RCM

- **Ruthenium Metathesis Catalysts Kit I**

Kit consists of 9 samples of Grubbs 1st and 2nd generation catalysts

**Catalog No.** 687944

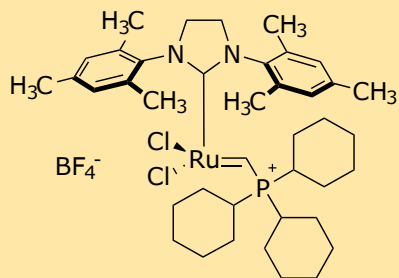
- **Grubbs Catalyst® C859**

Active as low as -50 °C, which enables direct observation of reaction intermediates

**Molecular Weight:** 858.67

**CAS:** 832146-68-6

**Catalog No.** 707988



# Easy Access to Grubbs Catalyst® Technology

We hope that this guide and our collaboration with Umicore will inspire you to reach new frontiers in chemical synthesis. Enjoy unparalleled access to Grubbs Catalyst® technology, and experience the many benefits of our exclusive partnership.

- Secure supply chain of metathesis catalysts from milligram to multi-kilogram volumes
- Rapid delivery of R&D quantities
- Access to technical expertise, supported by Nobel Laureate Robert Grubbs
- Umicore license rights included in listed product price

**Discover more:**

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