

# TCIMAIL

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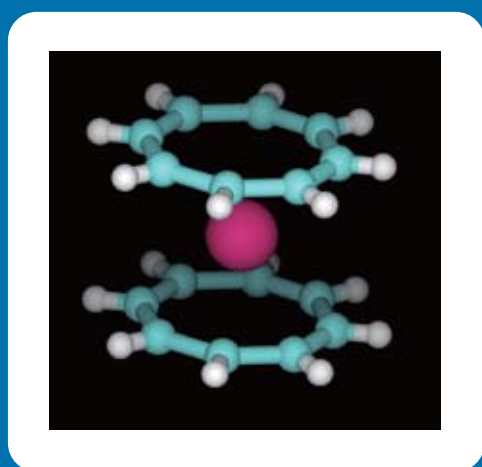
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## Chemistry between Carbon and Other Elements

Kentaro Sato

Numerous research articles are published every day in the field of organic chemistry. Come to think of it, in the genre that deals with only one element - carbon, considering such high pace of publication, one might wonder how researchers manage not to run out of new ideas. Apparently, the possibility of carbon chemistry has a large space left to be explored after hundreds of years of history, and the horizon even continues to expand.

In the CAS database, the largest chemical database in the world, close to 70 million compounds are registered. Of those, 80 percents are carbon-based organic compounds. Within the periodic table consisting of more than 100 elements, carbon has a special status which may be called the “king of all elements.”

There are several reasons why carbon is such a special element, and one of them is because of its ability to form stable bond(s) with a number of other “heteroatom” elements. Carbon forms bonds with not only common non-metal elements such as hydrogen, oxygen, nitrogen, sulfur, phosphorus, and halogens, but also with most metallic elements. Many of the compounds based on carbon-heteroatom bond are common reagents found in the shelves of chemistry laboratories.

Without needing to mention such prime examples as Grignard reagent, hydroboration, and Wittig reaction, carbon-heteroatom bond has always been trailblazing the development of organic chemistry. Even relatively less familiar heavy metal elements such as hafnium, rhenium, and bismuth have been utilized for organic synthetic reactions. Molybdenum and ruthenium carbenes used to be somewhat exotic compounds until not too long ago, but became mainstream regulars after the advent of Grubbs olefin metathesis catalyst. The development of new chemical

bond is always the most important frontier of chemistry, and as such, it continuously draws a great deal of interest and energy from scientific community.

### Organic Noble Gas Compounds

The synthesis of the first xenon-containing compound XePtF<sub>6</sub> by Neil Bartlett in 1962 is a renowned accomplishment listed in many textbooks. Since then, xenon compounds of different oxidation numbers (II, IV, VI, VIII) have been known to date and many of those are fluorides and oxides. Among them, for example, XeF<sub>2</sub> is a useful (and commercially available) fluorination reagent.

Xenon is also known to form a bond with carbon. It has a surprisingly long history, with the first preparation of its kind reported back in 1989 [1]. For Xe(II) compound, C<sub>6</sub>F<sub>5</sub>XeF has been used to synthesize several organic xenon compounds including Xe(C<sub>6</sub>F<sub>5</sub>)<sub>2</sub> [2].

In 2000, the first organic xenon(IV) compound was synthesized [3]. For example, [C<sub>6</sub>F<sub>5</sub>XeF<sub>2</sub>]BF<sub>4</sub> was made by the reaction between XeF<sub>4</sub> and C<sub>6</sub>F<sub>5</sub>BF<sub>2</sub>. Reported to act as a strong fluorinating agent, this compound perhaps has an interesting chance to develop into a reagent.

For krypton, which is one size smaller family member of xenon, it is known that photo irradiation in the presence of acetylene under low temperature conditions produces HC≡CKrH [4]. There might be similar possibility for radon, but so far only Rn(II) fluoride is known and the synthesis of organic radon compound has not been realized. How about argon and neon? To find out whether they can react with carbon, there is probably a competition going on among laboratories around the world.

## Hot Organic Compounds

High sociability of carbon extends to the realm of radioactive elements. For example, short-lived astatine (having a half-life of maximum 8.1 hours) forms organic compounds such as  $C_6H_5At$ . Actinide elements such as uranium, neptunium, plutonium, and americium also complex with cyclooctatetraene to form sandwich-shaped molecules commonly called “hot sandwiches” (picture shown below) [5]. These complexes are rare molecules having a symmetry feature known as  $D_{8h}$ . For uranium, there is  $Cp_4U$  complex, in which four cyclopentadienyl ligands are bonded to the uranium in tetrahedral fashion [6]. Big atoms have surprising properties indeed.

To the best of my literature search, the heaviest element that forms a bond with carbon is einsteinium with atomic number 99. In 2005, the synthesis of an alkene-einsteinium complex was reported [7]. Among smaller elements than this, there are only a few which are not known to bond with carbon; the examples are some of noble gases like helium, neon, and argon, and extremely short-lived species such as francium.

By the way, if the partner is not limited to carbon, the heaviest element ever prepared as a compound is hassium with atomic number 108. In 2002, hassium compounds including  $HsO_4$  were synthesized from just seven atoms and their properties have been studied [8]. The isotope used in the study had a half life of only 11 seconds, which tells us about the incredibly high level of experimental sophistication. Considering that, there may still be rooms in the list of elements that potentially make a bond with carbon.

## The Element Not Found in The Periodic Table

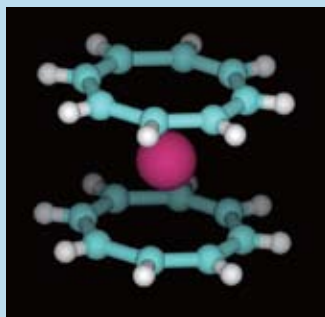
In 2008, an element called muonium emerged as a new chemical partner of carbon. Most people probably have never heard of this “element”, however, because it is not found anywhere in the periodic table. Muonium is not exactly an element which consists of proton, neutron, and electron.

There is an elemental particle called muon. This particle is 207 times heavier than electron and can have either a positive or a negative charge. A positively charged muon captures an electron upon encounter to form a hydrogen-like “atom” called muonium, which is considered one of the “exotic atoms” having neither proton nor neutron.

P. W. Percival and his group used muonium as an equivalent of hydrogen radical while studying the reactivity of carbon-silicon double bonds [9]. In this study, a compound in which muonium is bonded to carbon was observed. As a research method of organic chemistry, this is a very atypical but impressive approach.

You may wonder how stable the carbon-muonium bond is. Muon itself exists with an average longevity of only 2.2 microseconds, therefore, the organic muonium species is destined to vanish in split seconds literally.

It is surprising that any meaningful data can be collected in that instantaneous period of time, but according to the experts in the field of elemental particle research, muonium actually belongs to the group of long-lived particles and its analysis is relatively easy. For example, positronium (an exotic atom composed of an electron and a positron) is also known to form a compound but it lives for nanoseconds, therefore, its detection is “slightly difficult.” The physical chemists certainly have a very different perception about time, but this kind of interdisciplinary laboratory should be a great place where fascinating researches are born.



“Hot sandwiches”

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## Introduction of the author :

### Kentaro Sato

[Brief career history] He was born in Ibaraki, Japan, in 1970. 1995 M. Sc. Graduate School of Science and Engineering, Tokyo Institute of Technology. 1995-2007 Researcher in a pharmaceutical company. 2007-Present Freelance science writer. 2009-2012 Project assistant professor of the graduate school of Science, the University of Tokyo.

[Specialty] Organic chemistry

[Website] The Museum of Organic Chemistry <<http://www.org-chem.org/yuuki/MOC.html>>

## Technical Glossary

### Carbene complex

*p.2 "Chemistry between Carbon and Other Elements"*

Carbene complexes are organometallic complexes with the carbene coordinated on the carbon atom to the metal center. There are two types which can be categorized as Fischer type carbene complexes and Schrock type carbene complexes.

Fischer carbene complexes show electrophilicity because the center metal is in an electron poor state. Therefore, the reactivity of it is similar to carbonyl compounds in which nucleophilic substitution of it with various nucleophiles proceeds. In addition one of substituents has to be an alkoxy group because of its electronic structure.

The chemical character of Schrock type carbene complexes is nucleophilic because of an electron rich state at the metal center. It shows especially high reactivities towards carbon-carbon and carbon-heteroatom multiple bonds. As typical chemical examples of Schrock type carbene complex usage, olefin metathesis and carbonyl olefination are well known. These synthetic characteristics are very useful so many suppliers have Schrock type carbene complexes as reagents.

### Olefin metathesis

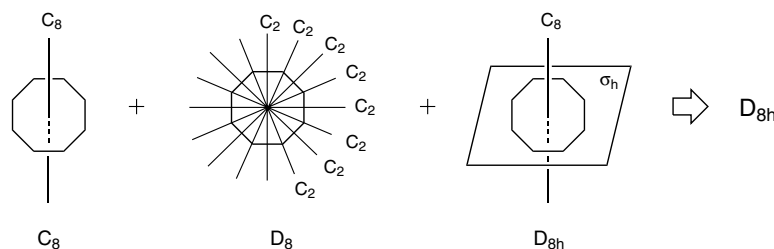
*p.2 "Chemistry between Carbon and Other Elements"*

Olefin metathesis is a chemical reaction between double bonds of two separate olefin units in which double bonds exchange with each other to form two new olefin units. A Schrock type carbene complex is usually used for such a synthesis and Mo, Ru and Ti metals are generally used as active metals. The bulkiness of substituents on the olefins affects their reactivity in olefin metathesis, so the development of new carbene complexes showing higher reactivity has been studied since the 1990's.

### $D_{8h}$ symmetry

*p.3 "Chemistry between Carbon and Other Elements"*

A molecule having a  $C_n$  principal rotation axis and its perpendicular  $n$  elements of  $C_2$  rotation axes is in the  $D_n$  group. In addition, a molecule having a  $\sigma_h$  mirror perpendicular to the principal axis shows  $D_{nh}$  symmetry. The molecule satisfying the conditions in the figure drawn below is in  $D_{8h}$  group.



## Mendeley, A Reference Manager and Social Network

Soichi Tokizane

Library and Information Science, Faculty of Letters, Aichi University

### 1. Mendeley for Reference Management

Most researchers store copies of reference articles in the form of PDF today. PDF copies occupy less space than paper copies, but it is harder to dig out necessary articles. Thus researchers often use so-called reference managers to organize references which they collected.

Commercially-available reference managers such as EndNote and RefWorks have been quite popular among researchers, while a new-comer, Mendeley, is mentioned more and more recently. Mendeley, a free tool, consists of Mendeley Desktop for PC and Mac, and Mendeley Web for browsers. Through synchronization, one can view stored articles at work, at home, or even while traveling on your iPad.

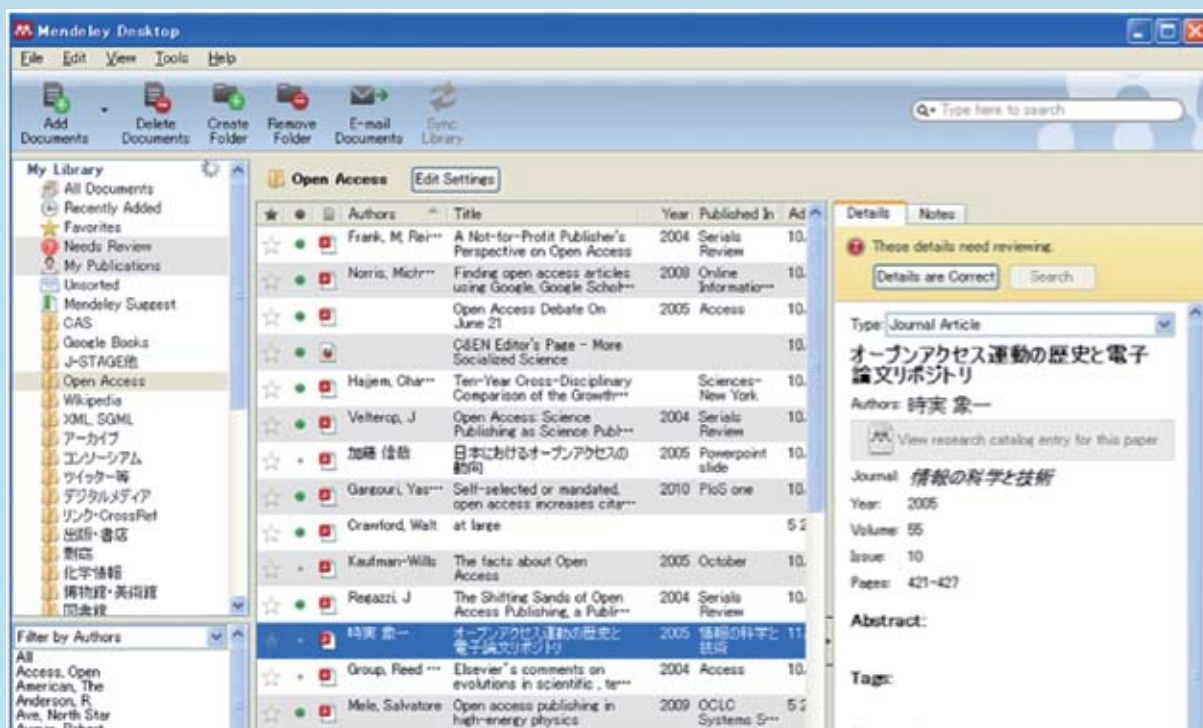


Figure 1. Mendeley Desktop



Figure 2. Mendeley Web

To register your references, you may simply drag-and-drop your PDFs to the Mendeley Desktop. It will analyze those PDFs, extract necessary data, check the Web for correct bibliographic data, and register them into your PC/Mac. Mendeley Web has a registration tool, Web Importer, which works similarly. After you find a document via a web search, click "Web Importer" in your Bookmark. Registered PDFs are stored in your library under appropriate category folders which you defined. You may sort PDFs by author names or search by keywords. You can store any

documents in addition to PDF, such as Word documents, or PowerPoint slides.

Japanese documents had been difficult to register, because Mendeley had difficulty in analyzing Japanese-language PDFs. Now a web tool, "Japanese Articles to Mendeley", helps register Japanese articles.

You can even create reference lists using the MS Word Plugin from your Mendeley database. There are already reference templates for more than 1000 journals.



Figure 3. "Japanese Articles to Mendeley"

## 2. Mendeley as a database

Mendeley Web has a search box named, "Paper", though which you may search relevant articles from the vast collection of documents which have been registered by Mendeley users worldwide. If the articles found are open access, you may be able to store such PDFs directly to your library. If not, you can store bibliographic data at least. Any references you have registered to your library are thus a part of this global collection. If you are a researcher of a private corporation, you may want to use Mendeley Desktop without synchronization to protect research secrets.

## 3. Mendeley as a social network

Mendeley is a social network. You are encouraged to register your profile with Mendeley, create a research group network, and share your comments and references. This is a new way of conducting research globally.

Mendeley is not only a reference manager, but also a powerful research tool. Please try Mendeley and enjoy using it.

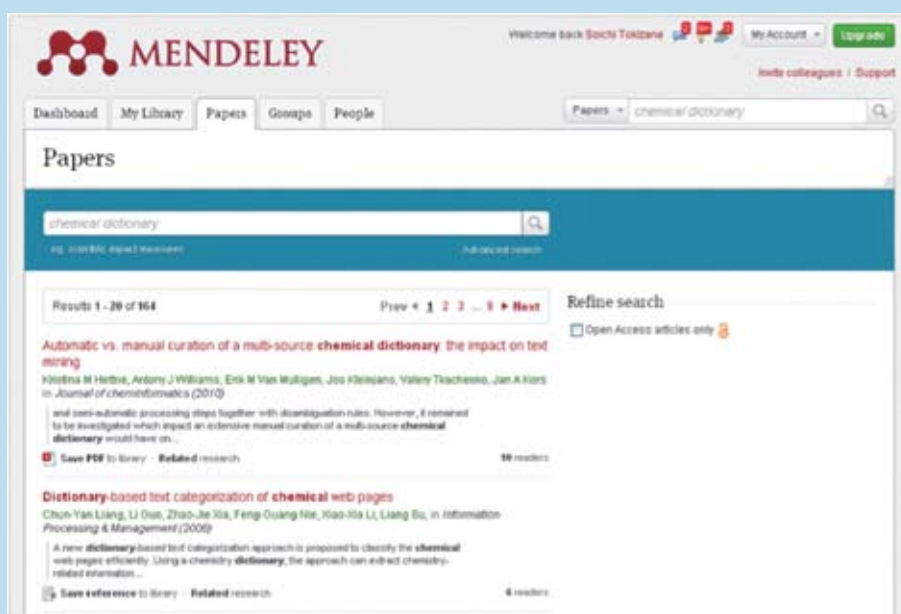


Figure 4. Search result of "Chemical Dictionary" at Mendeley Web



Figure 5. The author's profile at Mendeley



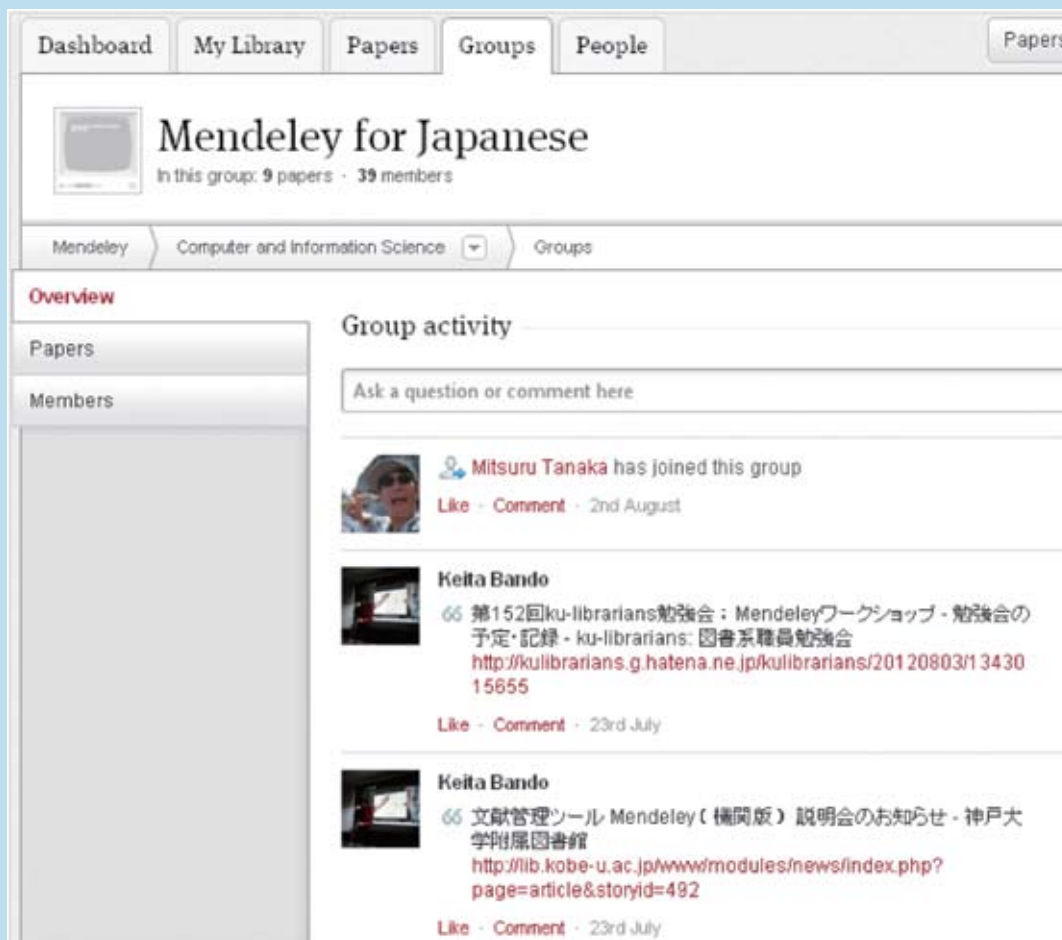


Figure 6. Mendeley Group

### Introduction of the author :

#### Soichi Tokizane

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[Biography] Master degree in chemistry at the Faculty of Science, the University of Tokyo. Job history includes Toray, the Japan Association for International Chemical Information (JAICI), Chemical Abstracts Service (CAS), the Japan Science and Technology Agency (JST). A member of the Advisory Board on Science and Technology Literature of the National Diet Library.

Authored books such as "Searching chemical literature and database in the internet era", "Using SciFinder", and "Internet for Science Students".

Majors in chemical information, and library and information science.

## More ways to use reagents

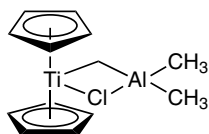
## Ring-Closing Metathesis Using Tebbe or Petasis Reagent

Haruhiko Taguchi

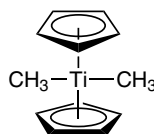
Tokyo Chemical Industry, Co. Ltd.

About 22,000 reagents are available from TCI. It is considered that each of them has various usages for its research field and almost all of them would be used as major usages such as synthetic reagents, building blocks, electronic materials, life science materials and so on. On the other hand, a number of reagents have been used in a minor but unique synthetic manner. In this column, we will present another usage of reagents from the viewpoint of the reagent company.

In this issue, I pick Tebbe and Petasis reagents. It is well known that Tebbe and Petasis reagents are methylenation reagents of carbonyl compounds and they are available from TCI. Most users actually buy these reagents for such a usage. We can see by the strategies in many articles of organic synthesis that methylenation using either one and following with Grubbs reagent promoted ring-closing metathesis (RCM) is very efficient in total synthesis. I'm sure that Tebbe and Petasis, and of course, Grubbs reagents are essential reagents for synthesis of macrocyclic compounds because terminal olefins can be easily obtained from Tebbe or Petasis reagents.



Tebbe Reagent

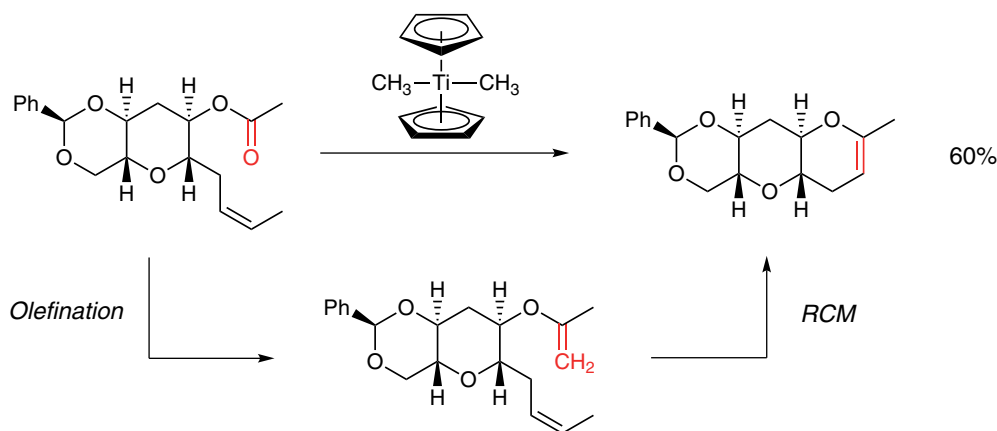


Petasis Reagent

We had performed macrocyclizations for the synthesis of a large number of cyclic compounds by Yamaguchi-macrolactonization and Corey-Nicolaou macrolactonization. And then, the intramolecular Mizorogi-Heck reaction had been developed and applied for these syntheses. In the 90's, Ru- and Mo-promoted RCM had been developed, and now this synthetic procedure has been one of the most useful methods for the synthesis of the large cyclic compounds.<sup>1)</sup>

Meanwhile, carbene complexes formed from Ru or Mo metals have been well used for RCM because these carbene complexes have high affinities and reactivities for terminal olefins especially. While Tebbe and Petasis reagents are the precursors of Ti-carbene complexes, in situ generated Ti-methylidene complexes react with olefins to proceed to olefin metathesis with similarities to Ru- or Mo-carbene complex-promoted olefin metathesis.

However, in the case of Ti-carbene complexes, the reactivity is different from other metal-carbene complexes. Ti-carbene complex shows a higher affinity for the carbonyl group compared with the olefin moiety and this result impresses us as this reagent can be used for carbonyl olefination. Actually, Tebbe and Petasis reagents are the reagent for carbonyl olefination, but they can be also used for olefin metathesis. I considered how can use these reagent for olefin metathesis similar to Grubbs reagent and were there some chemists who had similar idea? So I searched related references and found that K. C. Nicolaou and his coworkers studied the reactivities of RCM using Tebbe and Petasis reagents.<sup>2)</sup> I will summarize the contents of these articles.



Nicolaou *et al.* studied RCM of marine natural products having a cyclic polyether structure. As a result, 6- and 7-membered cyclic polyethers were successfully synthesized by using Tebbe or Petasis reagents. However, this synthetic method was effective only when fixed-structure substances such as cyclic polyethers were employed. No desired products were afforded when substances having high flexibility such as acyclic polyethers were used. These results impress on us that the Grubbs reagent is the great reagent which can be used for various substances in RCM.

Recently, RCM using  $\text{CH}_3\text{CHBr}_2\text{-TiCl}_4\text{-Zn}$  system has been reported.<sup>3)</sup> This reagent proceeds formally with the same reaction as other titanium reagents and even more, it shows high reactivity against Tebbe and Petasis reagents. This is the first report that acyclic substances of a diene can be used for titanium mediated RCM. Organotitanium reagents have been developed step by step and they may be used as one of the methods for macrocyclization in the future.

As described above, Tebbe and Petasis reagents are a powerful tool for methylenation of carbonyl compounds and they have another usage for RCM. Though this usage is limited for substances, I think these reagents are attractive and unique reagents. You can choose various organotitanium reagents from TCI for the study of organic synthesis.

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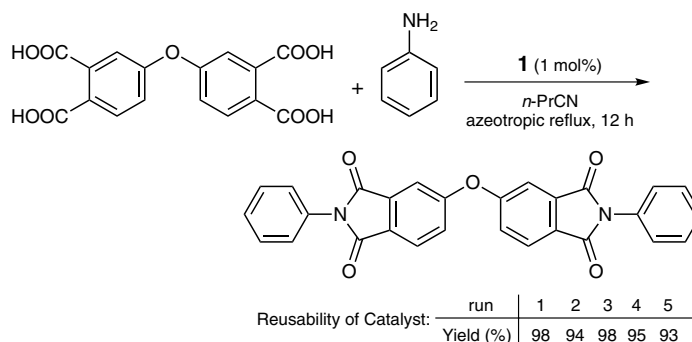
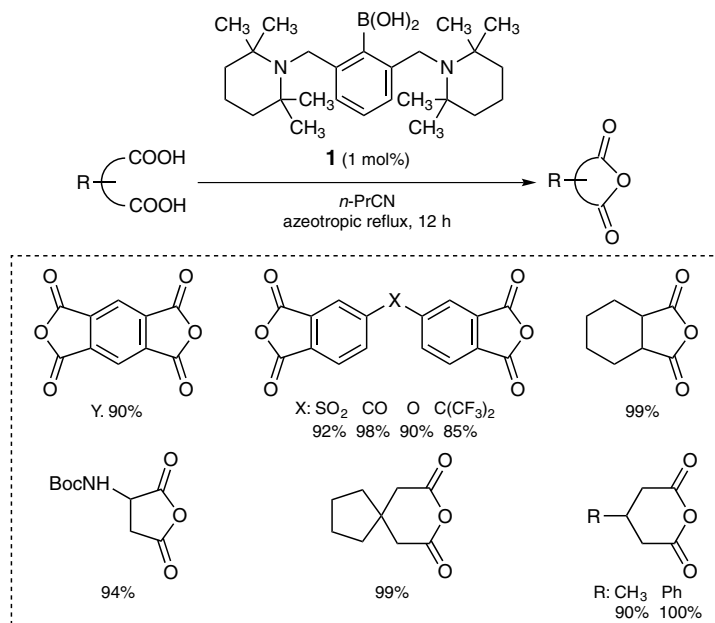
## Related Products

C1411	Tebbe Reagent (ca. 0.5mol/L in Toluene)	5mL
D4100	Dimethyltitanocene (5% in Tetrahydrofuran/Toluene)	100g
T0616	Titanocene Dichloride	5g, 25g
T2052	Titanium(IV) Chloride (14% in Dichloromethane, ca. 1.0mol/L)	100mL

## Dehydrative Intramolecular Condensation Catalyst for the Synthesis of Carboxylic Anhydrides

B3927 2,6-Bis[(2,2,6,6-tetramethyl-1-piperidinyl)methyl]phenylboronic Acid (contains varying amounts of Anhydride) (**1**)

200mg, 1g



**1** is a hybrid catalyst developed by Ishihara *et al.*, which has Lewis acid and Brønsted base moieties in a molecule. A small amount of **1** smoothly catalyzes the dehydrative intramolecular condensation of dicarboxylic acids, such as phthalic acid, to afford the corresponding carboxylic anhydrides in high yields under mild conditions. In addition, it has been also reported that a diimide can be directly synthesized from a tetracarboxylic acid and a primary amine as a model reaction for the synthesis of polyimides.

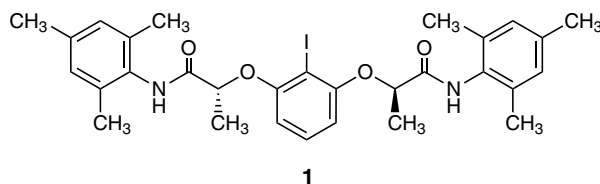
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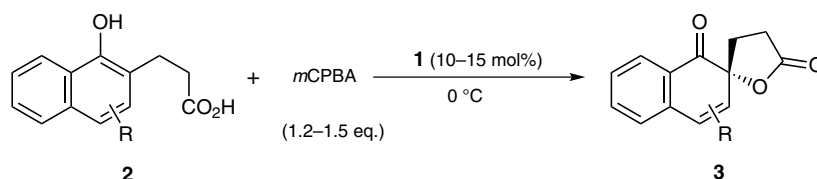
## Precursor of the Chiral Hypervalent Iodine Catalyst

I0807 (R,R)-2-Iodo-1,3-bis[1-(mesitylcarbamoyl)ethoxy]benzene (1)

200mg



(*R,R*)-2-Iodo-1,3-bis[1-(mesitylcarbamoyl)ethoxy]benzene (**1**), which was developed by Ishihara *et al.*, is a precursor of the chiral hypervalent iodine catalyst, and its utility for the enantioselective intramolecular oxidative coupling reaction (Kita Spirolactonization) has been reported. The chiral hypervalent iodine(III) species, which is generated *in situ* from **1** and *m*CPBA as a co-oxidant, catalyzes the enantioselective spirolactonization of 1-naphthol derivatives **2** to afford the corresponding spirolactones **3** with high enantioselectivities. This reaction is environmentally benign because it does not have to use any toxic metal reagents. Since spirolactones are used as pharmaceutical intermediates, it is expected to be applied to pharmaceutical synthesis.



R	Conditions	Y. (%)	ee (%)
H	CHCl <sub>3</sub> , 18 h	60	92
4-Me	CHCl <sub>3</sub> /CH <sub>3</sub> NO <sub>2</sub> <sup>a)</sup> , 17 h	59	84
4-Br	CHCl <sub>3</sub> , 16 h	67	85 (98) <sup>b)</sup>
4-COPh	CHCl <sub>3</sub> /CH <sub>3</sub> NO <sub>2</sub> <sup>a)</sup> , 16 h	94	83 (>99) <sup>b)</sup>
6-OMe	CHCl <sub>3</sub> /CH <sub>3</sub> NO <sub>2</sub> <sup>a)</sup> , 18 h	40	87

a) CHCl<sub>3</sub>/CH<sub>3</sub>NO<sub>2</sub> (2:1, v/v), b) After a single recrystallization

Typical Procedure<sup>1b)</sup> :

A solution of **2a** (R=H) (216 mg, 1.0 mmol), **1** (92.2 mg, 0.15 mmol, 15 mol%) and *m*CPBA (269 mg, 1.2 mmol, 1.2 eq.) in CHCl<sub>3</sub> (50 mL) is stirred at 0 °C. After 18 h, the resulting mixture is poured into aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (20 mL) and aqueous NaHCO<sub>3</sub>, and extracted with CHCl<sub>3</sub> (2 times). The organic layers are dried over anhydrous MgSO<sub>4</sub> and solvents are removed in vacuo. The residue is purified by flash column chromatography on silica gel (hexane–EtOAc = 10:1 to 4:1) to give **3a** (129 mg, 0.6 mmol) in 60% yield (92%ee).

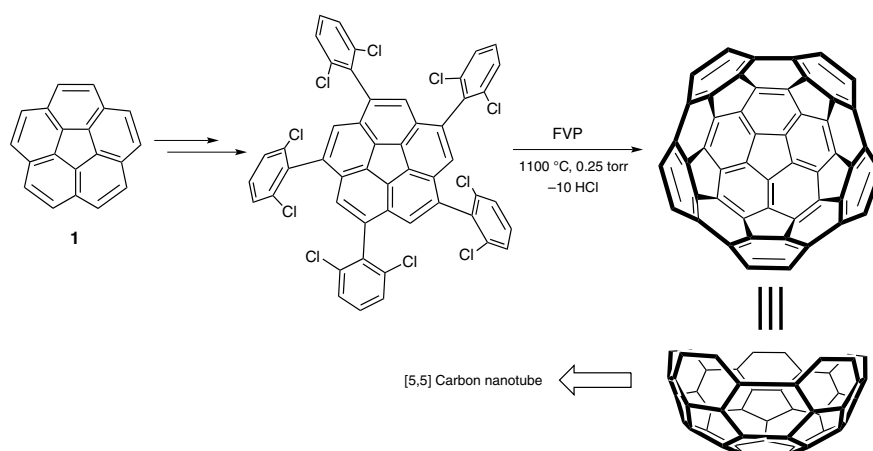
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## Application to the Synthesis of [5,5] Carbon Nanotube

C2572 Corannulene (1)

20mg, 100mg



Corannulene (1) is an aromatic polycyclic hydrocarbon with a unique bowl-shaped structure. Scott *et al.* have reported the synthesis of the polyarene by flash vacuum pyrolysis (FVP) using 1. This polyarene compound is an equivalent of a short end-capped [5,5] carbon nanotube, therefore, the chemical synthesis of a [5,5] carbon nanotube is expected by using the polyarene compound as a template to grow a nanotube.

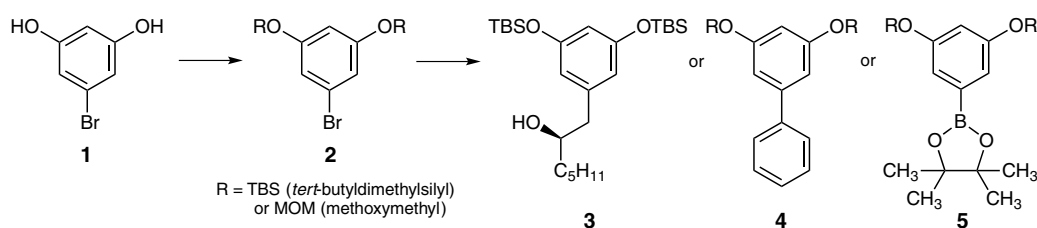
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A short, rigid, structurally pure carbon nanotube by stepwise chemical synthesis  
L. T. Scott, E. A. Jackson, Q. Zhang, B. D. Steinberg, M. Bancu, B. Li, *J. Am. Chem. Soc.* **2012**, *134*, 107.

## A Valuable Synthetic Intermediate of 5-Substituted Resorcinols

B3731 5-Bromoresorcinol (1)

1g, 5g



5-Bromoresorcinol (1) is a valuable intermediate for the preparation of a number of 5-substituted resorcinols, such as 3, 4 and 5. The compound 3 is the synthetic intermediate of (–)-berkelic acid.<sup>1)</sup> Compounds 4 are the building blocks of biphenyl compounds<sup>2)</sup> and compounds 5 are 3,5-disubstituted arylboronic pinacol esters for Suzuki-Miyaura coupling.<sup>3)</sup>

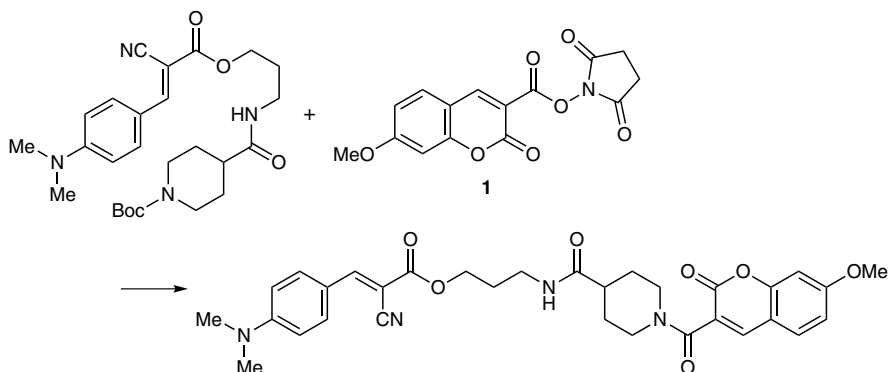
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## Fluorescent Label

S0867 *N*-Succinimidyl 7-Methoxycoumarin-3-carboxylate (1)

100mg, 1g



Fluorescent label, *N*-succinimidyl 7-methoxycoumarin-3-carboxylate (1), has been applied to labeling not only for macromolecules such as proteins, but also for small molecules. One of the examples is introduced below.

It has been proposed that monitoring of biofluid viscosity could provide a diagnostic tool for the detection of diseases.<sup>1)</sup> Recently, a procedure for the synthesis of a ratiometric viscosity fluorescent sensor using 1 has been reported.<sup>2)</sup> In this report, the coumarin moiety was used as a viscosity-independent fluorophore, and *p*-aminocinnamoyl dye as a viscosity-dependent fluorophore.

### References

- 1) The viscosity of human blood plasma; its measurement in health and disease  
J. Harkness, *Biorheology* **1971**, 8, 171.
- 2) Synthesis and use of an in-solution ratiometric fluorescent viscosity sensor  
D. Fischer, E. A. Theodorakis, M. A. Haidekker, *Nat. Protocols* **2007**, 2, 227.

## Chemiluminescence Enhancer

B3910 4-Bromophenol (1)

1g, 5g

I0840 4-Iodophenol (2)

1g, 5g



In research for life-science, one of the frequently used chemiluminescence (CL) reagents is luminol. Luminol reacts with hydrogen peroxide in the presence of a catalyst, for example, enzymes, metals such as iron, or metal-complexes to emit strong light. In the case of luminol, blue light with a wavelength of 460 nm is emitted.

Since Thorpe *et al.* reported that phenolic compounds such as 4-iodophenol enhance CL by a peroxidase-luminol-hydrogen peroxide reaction,<sup>1)</sup> many improved methods have been reported and are called enhanced chemiluminescence (ECL). ECL is one of the sensitive and rapid detection methods in medical and analytical biochemistry.<sup>2,3)</sup>

### References

- 1) Phenols as enhancers of the chemiluminescent horseradish peroxidase-luminol-hydrogen peroxide reaction: application in luminescence-monitored enzyme immunoassays  
G. H. G. Thorpe, L. J. Kricka, S. B. Moseley, T. P. Whitehead, *Clin. Chem.* **1985**, 31, 1335.
- 2) Chemiluminescent and bioluminescent methods in analytical chemistry  
L. J. Kricka, G. H. G. Thorpe, *Analyst* **1983**, 108, 1274.
- 3) Enhanced chemiluminescence enzyme immunoassay  
L. J. Kricka, G. H. G. Thorpe, R. A. W. Stott, *Pure Appl. Chem.* **1987**, 59, 651.

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