Handbook of Reagents for Organic Synthesis

Reagents, Auxiliaries, and Catalysts for C–C Bond Formation

Edited by

Robert M. Coates
University of Illinois at Urbana-Champaign

and

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This volume is dedicated to the memory of William G. Dauben, an inspiring mentor and forthright colleague. We acknowledge the authors of the original articles in the *Encyclopedia of Reagents for Organic Synthesis* whose work forms the large body of this new edition. We would like to thank Professor Jeremiah P. Freeman and *Organic Syntheses* for providing the original graphics for the *Organic Syntheses* procedures presented in Section 3C.

We appreciate the assistance of Shirley Pierson and Linda Hirsch for the reformatting and compilation of much of the introductory material.

Robert M. Coates and **Scott E. Denmark**

Editorial Board

Preface

As stated in its Preface, the major motivation for our undertaking publication of the *Encyclopedia of Reagents for Organic Synthesis* was "to incorporate into a single work a genuinely authoritative and systematic description of the utility of all reagents used in organic chemistry." By all accounts, this reference compendium has succeeded admirably in attaining this objective. Experts from around the globe contributed many relevant facts that define the various uses characteristic of each reagent. The choice of a masthead format for providing relevant information about each entry, the highlighting of key transformations with illustrative equations, and the incorporation of detailed indexes serve in tandem to facilitate the retrieval of desired information.

Notwithstanding these accomplishments, the editors have since recognized that the large size of this eight-volume work and its cost of purchase have often detered the placement of copies of the *Encyclopedia* in or near laboratories where the need for this type of information is most critical. In an effort to meet this demand in a cost-effective manner, the decision was made to cull from the major work that information having the highest probability for repeated consultation and to incorporate same into a set of handbooks. The latter would also be purchasable on a single unit basis.

The ultimate result of these deliberations is the publication of the *Handbook of Reagents for Organic Synthesis* consisting of the following four volumes:

Reagents, Auxiliaries, and Catalysts for C–C Bond Formation
edited by Robert M. Coates and Scott E. Denmark

Oxidizing and Reducing Agents
edited by Steven D. Burke and Rick L. Danheiser

Acidic and Basic Reagents
edited by Hans J. Reich and James H. Rigby

Activating Agents and Protecting Groups
edited by Anthony J. Pearson and William R. Roush

Each of the volumes contains a selected compilation of those entries from the original *Encyclopedia* that bear on the specific topic. Ample listings can be found to functionally related reagents contained in the original work. For the sake of current awareness, references to recent reviews and monographs have been included, as have relevant new procedures from *Organic Syntheses*.

The end product of this effort by eight of the original editors of the *Encyclopedia* is an affordable, enlightening set of books that should find their way into the laboratories of all practicing synthetic chemists. Every attempt has been made to be of the broadest synthetic relevance and our expectation is that our colleagues will share this opinion.

Leo A. Paquette
Columbus, Ohio USA

Introduction

Every practicing organic chemist recognizes the critical importance of selecting the most appropriate reagent and reaction conditions for executing a given chemical transformation. While a working knowledge of the most common reagents employed in organic synthesis is essential for every synthetic chemist, even the most dedicated practitioner can hardly claim fluency with the overwhelming array of inorganic and organic reagents now at his or her disposal. Moreover, the development of new and more selective reagents has accelerated exponentially and still constitutes one of the most vigorous areas of organic chemistry research. In recognition of this challenge, the Editors of the *Encyclopedia of Reagents for Organic Synthesis (EROS)* have selected approximately 500 of the most important and useful reagents employed in organic synthesis. In keeping with our goal to provide a concise, desktop reference work, we culled the most important and commonly used reagents from the over 3000 entries in the original *EROS*. These selections comprise the *Handbook of Reagents for Organic Synthesis* and cover the full range of chemical types and transformations. The *Handbook* is divided into four volumes that contain reagents of similar type and/or function to assist the user in rapidly locating a reagent of choice: *Reagents, Auxiliaries, and Catalysts for C–C Bond Formation; Oxidizing and Reducing Agents; Acidic and Basic Reagents*; and *Activation Agents and Protection Groups*.

This volume entitled *Reagents, Auxiliaries, and Catalysts for C–C Bond Formation* contains the largest and most diverse group of reagents by virtue of the broad scope of chemical reactions included in this general category. Two major types of reagents selected from the parent *EROS* are essential carbon-nucleophiles and carbon-electrophiles widely used to form C–C bonds in synthesis. The C-nucleophile group includes numerous organometallic reagents, carbanions, enolates, ylides, and their precursors. Familiar alkylating, acylating, and cyclopropanating reagents together with Michael acceptors and other electron-deficient olefins comprise a fundamental group of C-electrophiles. An indispensable subset of reagents for cycloadditions includes acetylenes, allenes, dienes, dienophiles, and ketenes. However, many other reagents extensively represented in this volume are critical participants in C–C bond forming reactions, which are not themselves incorporated into the products, such as catalysts, chiral auxiliaries, and selected heteroatom electrophiles. Particular attention was paid to the selection of the most important chiral catalysts and auxiliaries in recognition of the growing importance of enantioselective and diastereoselective transformations. Numerous transition-metal catalysts and stoichiometric metalloid reagents vital for C–C coupling reactions and cyclopropanations were deemed appropriate. While efforts were made to minimize duplication, a number of entries found in this volume have multiple applications and therefore appear in other volumes, e.g. 1,4-benzoquinone (see also *Oxidizing and Reducing Agents*), cerium trichloride (see also *Acidic and Basic Reagents*), chromium(II) chloride (see also *Oxidizing and Reducing Agents*), diazomethane (see also *Activating Agents and Protecting Groups*) and thexylborane (see also *Oxidizing and Reducing Agents*). On the other hand, some obvious redox reagents such as nickel(II) chloride, palladium(II) acetate and chloride, and manganese(III) acetate were chosen exclusively for this volume on account of their primary function in C–C bond formation. Although chlorotrimethylsilane, tri-*n*-butylchlorostannane, (*R*)- and (*S*)-menthyl *p*-toluenesulfinates, *p*-toluenesulfonyl azide, and *p*-toluenesulfonylhydrazide are not themselves used for C–C bond formation, their crucial role in the synthesis of C–C bond forming reagents provided a compelling rationale for their inclusion in this volume. In contrast, reagents such as the isomeric butyllithiums and pyrrolidine, while obviously closely associated with the generation of C–C bond forming reagents, are assigned exclusively to *Acidic and Basic Reagents* in recognition of their primary function as bases.

To familiarize the user with the spectrum of reagents contained in this volume and to organize the pertinent reference material, we have subdivided the 203 featured reagents into 22 classes based on their chemical structures. Clearly many reagents are multi-functional and could be logically assigned to several different classes. In many cases the class assignment was based upon the functionality that would normally be retained in the product of their common synthetic applications. Some examples are ethyl bromozincacetate (Class 9: "Enolates, Homoenolates, and Dicarbonyl Compounds"), lithium bis[dimethyl(phenyl)silyl]cuprate (Class 18: "Silicon and Tin Reagents"), and trifluoromethyltrimethylsilane (Class 11: "Halo Compounds"). All but one (nitromethane) of the designated "C_1 Reagents" in Class 3 are carbonyl compounds commonly utilized as electrophilic reagents to introduce oxygen functionality in C–C bond forming reactions (e.g. formaldehyde and *N,N*-dimethylformamide). On the other hand, the common electrophilic C_1 reagents used to introduce nitrogen functionality are found in Class 12: "Imines, Iminium ions, and Amide Acetals" (e.g. formaldehyde-dimethlyamine and dimethylformamide acetal). These classifications should afford conceptual and organizational guidance for the users. It is worth while to remind readers that although headings designate a single compound, often related reagents (e.g. other esters, enantiomers of chiral reagents) are also covered. The reagent articles are arranged alphabetically in the volume, as they are in the original *EROS*. In addition, the Table of Contents also shows the class number to aid readers in finding reagents of similar structure and function.

1. Acetylene and Allenes
2. Aluminum and Boron Reagents
3. C_1 Reagents
4. Chiral Auxiliaries and Reagents
5. Copper Reagents
6. Cyano, Isocyano, and Isocyanato Reagents
7. Diazo, Hydrazido, and Azido Reagents
8. Dienes, Dienophiles, and Michael Acceptors
9. Enolates, Homoenolates, and Dicarbonyl Compounds
10. Epoxides
11. Halo Compounds
12. Imines, Iminium Ions, and Amide Acetals
13. Ketenes, Ketene Acetals, and Ortho Esters
14. Lithium and Magnesium Reagents
15. Nickel Reagents
16. Palladium Reagents
17. Phosphorus Reagents
18. Silicon and Tin Reagents
19. Sulfur Reagents
20. Titanium Reagents
21. Transition Metal and Lanthanide Reagents
22. Zinc Reagents

Each of the reagent entries appears almost verbatim from the original work (*EROS*). However, to make this new work as current as possible and to provide additional, useful information, the section Editors have compiled lists of recent reviews (1992- and 1998) in the general areas of each Volume. In addition, a collection of recent procedures from *Organic Syntheses* (*Vol. 70–75*) that feature preparations, reactions or applications pertinent to the content of this volume is provided in the front material. The reagent classes have been used to good advantage for the organization of this additional material that appears after the Table of Contents. The recent reviews and selected *Organic Syntheses* procedures are sorted and presented under the appropriate rubric of the 22 reagent classes. The Editors have taken a liberal view in the selection of recently published reviews, chapters, and monographs concerning important C–C bond forming methods related to each reagent class, whether or not the specific reagents appear in this volume. Furthermore, the Editors have culled out only the most relevant chemical transformations from multistep *Organic Syntheses* procedures. Finally, while the bulk of the entries are directly reproduced from *EROS*, they now all contain a **Related Reagents** section that guides the reader to reagents of similar structure or function that can be found either in the other volumes or in the original work.

The Editors of the *Handbook* are pleased with the content and organization of each of the four volumes in this set and we hope that your chemical enterprises will benefit from the efforts of the original researchers, authors and editors who have contributed to the creation of this valuable new resource.

Robert M. Coates
University of Illinois at Urbana-Champaign

Scott E. Denmark
University of Illinois at Urbana-Champaign

Contents

* Numbers in bold indicate the class number to within the Reagent belongs.

CLASS 1 Acetylenes and Allenes

A. Reagents

Acetylene
Bis(trimethylsilyl)acetylene
Ethoxyacetylene
Lithium Acetylide
1-Methyl-1-(trimethylsilyl)allene
Trimethylsilylacetylene

B. Reviews (1992–1998)

(1) Gleiter, R. and Kratz, D. Conjugated Enediynes-An Old Topic in a Different Light. *Angew. Chem., Int. Ed. Engl.* **1993**, *32*, 842–45.

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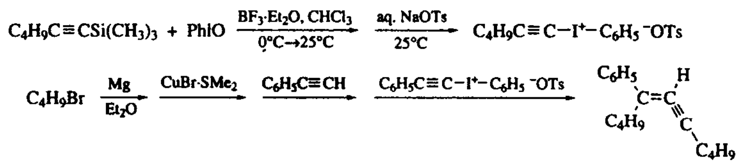
C. Organic Syntheses Procedures (Vols. 70–75)

Authors	Citation	Title
Peter J. Stang and Tsugio Kitamura	*Org. Synth.* **1992**, *70*, 215.	ALKYNYL(PHENYL)IODONIUM TOSYLATES: PREPARATION AND STEREOSPECIFIC COUPLING WITH VINYLCOPPER REAGENTS. FORMATION OF CONJUGATED ENYNES. 1-HEXYNYL(PHENYL)-IODONIUM TOSYLATE AND (*E*)-5-PHENYLDODEC-5-EN-7-YNE



CLASS 2 Aluminum and Boron Reagents

A. Reagents

Allenylboronic Acid
B-Allyldiisopinocampheylborane
9-Borabicyclo[3.3.1]nonane Dimer
Di-*n*-butylboryl Trifluoromethanesulfonate
Diisopinylcampheylboron Trifluoromethanesulfonate
Diisopropyl 2-Crotyl-1,3,2-dioxaborolane-4,5-dicarboxylate
Thexylborane
Triethyaluminum

Triethylborane
Trimethylaluminum

B. Reviews (1992–1998)

(1) Lohray, B. B. and Bhushan, V. Oxazaborolidines and Dioxaborolidines in Enantioselective Catalysis. *Angew. Chem., Int. Ed. Engl.* **1992**, *31*, 729–30.

(2) Deloux, L. and Srebnik, M. Asymmetric Boron-Catalyzed Reactions. *Chem. Rev.* **1993**, *93*, 763–84.

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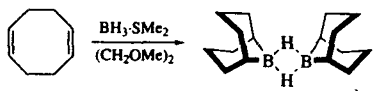
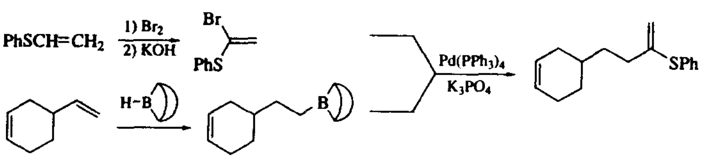
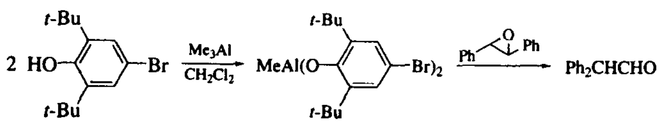
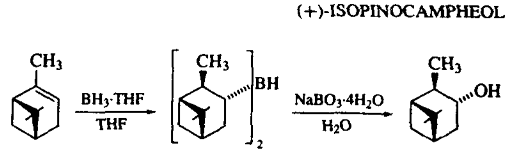
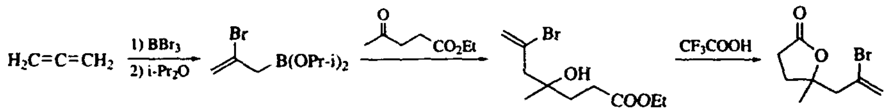
C. Organic Syntheses Procedures (Vols. 70–75)

Authors	Citation	Title
John A. Soderquist and Alvin Negron	*Org. Synth.* **1992**, *70*, 169.	9-BORABICYCLO[3.3.1]NONANE DIMER
		
Tatsuo Ishiyama, Norio Miyaura, and Akira Suzuki	*Org. Synth.* **1992**, *71*, 89.	PALLADIUM(0)-CATALYZED REACTION OF 9-ALKYL-9-BORABICYCLO[3.3.1]-NONANE WITH 1-BROMO-1-PHENYLTHIOETHENE: 4-(3-CYCLOHEXENYL)-2-PHENYLTHIO-1-BUTENE
		
T. Ooi, K. Maruoka, and H. Yamamoto	*Org. Synth.* **1993**, *72*, 95.	REARRANGEMENT OF *trans*-STILBENE OXIDE TO DIPHENYLACETALDEHYDE WITH CATALYTIC METHYLALUMINUM BIS(4-BROMO-2,6-DI-*tert*-BUTYLPHENOXIDE)
		
George W. Kabalka, John T. Maddox, Timothy Shoup, and Karla R. Bowers	*Org. Synth.* **1995**, *73*, 116.	A SIMPLE AND CONVENIENT METHOD FOR THE OXIDATION OF ORGANOBORANES USING SODIUM PERBORATE: (+)-ISOPINOCAMPHEOL
		

Shoji Hara and Akira Suzuki	*Org. Synth.* **1997**, *75*, 129	SYNTHESIS OF 4-(2-BROMO-2-PROPENYL)-4-METHYL-γ-BUTYROLACTONE BY THE REACTION OF ETHYL LEVULINATE WITH (2-BROMOALLYL) DIISOPROPOXYBORANE PREPARED BY HALOBORATION OF ALLENE



CLASS 3 C$_1$ Reagents

A. *Reagents*

Carbon Monoxide
Diethyl Carbonate
Dimethoxycarbenium Tetrafluoroborate
N,*N*-Dimethylformamide
Formaldehyde
Methyl Cyanoformate
Methyl Formate
Nitromethane
Paraformaldehyde

B. *Reviews (1992–1998)*

(1) Sonoda, N. Selenium-Assisted Carbonylation With Carbon Monoxide. *Pure Appl. Chem.* **1993**, *65*, 699–706.

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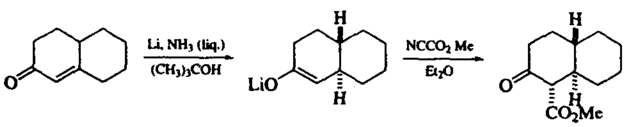
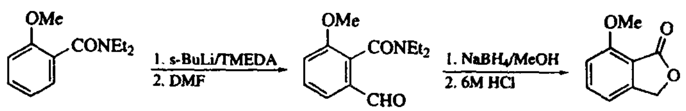
C. *Organic Syntheses Procedures (Vols. 70–75)*

Authors	Citation	Title
Simon R. Crabtree, Lewis N. Mander, and S. Paul Sethi	*Org. Synth.* **1992**, *70*, 256.	SYNTHESIS OF β-KETO ESTERS BY *C*-ACYLATION OF PREFORMED ENOLATES WITH METHYL CYANOFORMATE: PREPARATION OF METHYL (1α, 4aβ, 8aα)-2-OXO-DECAHYDRO-1-NAPHTHOATE



Authors	Citation	Title
X. Wang, S. O. deSilva, J. N. Reed, R. Billadeau, E. J. Griffen, A. Chan, and V. Snieckus	*Org. Synth.* **1993**, *72*, 163.	7-METHOXYPHTHALIDE



CLASS 4 Chiral Auxiliaries and Reagents

A. *Reagents*

(*S*)-1-Amino-2-methoxymethylpyrrolidine
(*S*)-4-Benzyl-2-oxazolidinone
(*R*)- and (*S*)-2,2′-Bis(diphenylphosphino)-1,1′-binaphthyl
(*R*)-2-*t*-Butyl-6-methyl-4*H*-1,3-dioxin-4-one
(*R*,*R*)-2-*t*-Butyl-5-methyl-1,3-dioxolan-4-one
(*R*)-(+)-Butyl 2-(*p*-Tolylsulfinyl)acetate
10,2-Camphorsultam
Chloro(cyclopentadienyl)-
bis[3-*O*-(1,2;5,6-di-*O*-isopropylidene-α-D-glucofuransoyl)]titanium
10-Dicyclohexylsulfonamidoisoborneol
(*S*)-(+)-2,5-Dihydro-2-isopropyl-3,6-dimethylpyrazine
(*R**,*R**) – α-2,6-Diisopropylbenzyloxy-5-oxo-1,3,2-dioxaborolane-4-acetic Acid
(*S*)-*N*,*N*-Dimethyl-*N*′-(1-*t*-butoxy-3-methyl-2-butyl)formamidine
2,2′-(Dimethylmethylene)bis(4-*t*-butyl-2-oxazoline)
trans-2,5-Dimethylpyrrolidine
(*R*,*R*)-1,2 Diphenyl-1,2-diaminoethane, *N*,*N*′-Bis[3,5-bis(trifluoromethyl)benzenesulfonamide]
1*R*,2*S*-Ephedrine
(*R*)- and (*S*)-Ethyl 3-Hydroxybutyrate
(*S*)- and (*R*)-Ethyl lactate
3-Hydroxyisoborneol
(*S*)-(+)- and (*R*)-(−)-Mandelic Acid
p-(*S*)- and (*R*)-Menthyl Toluenesulfinate
(*S*)-2-Methoxymethylpyrrolidine
(1*R*,2*S*) – *N*-Methylephedrine
α-Methyltoluene-2,α-sultam
(*R*)-Pantolactone
(2*R*,4*R*)-2,4-Pentanediol
(−)-8-Phenylmenthol
1,1,2-Triphenyl-1,2-ethanediol

B. *Reviews (1992–1998)*

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