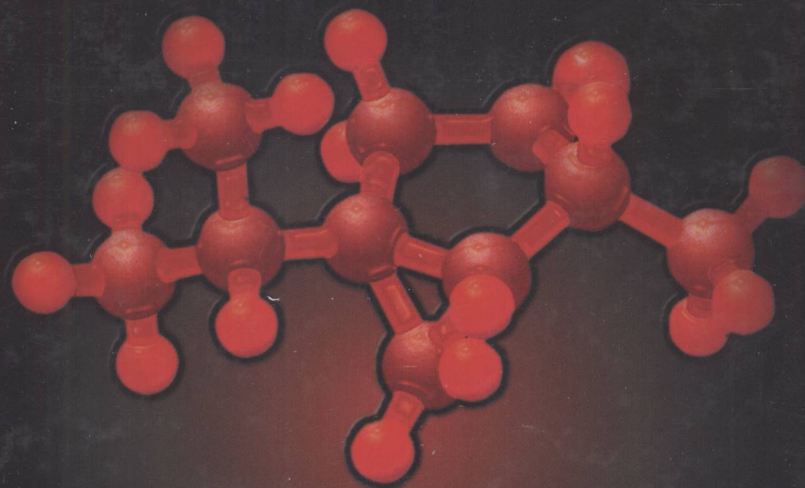




Handbook
of Reagents for
Organic Synthesis

**Reagents for
Glycoside, Nucleotide,
and Peptide Synthesis**

Edited by
David Crich

[v. 7]

Reagents for Glycoside, Nucleotide, and Peptide Synthesis

University of Illinois at Chicago, Chicago, IL, USA



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General Abbreviations

Ac	acetyl	DIEA	= DIPEA
acac	acetylacetonate	DIOP	2,3- <i>O</i> -isopropylidene-2,3-dihydroxy-1,4-bis-(diphenylphosphino)butane
AIBN	2,2'-azobisisobutyronitrile	DIPEA	diisopropylethylamine
Ar	aryl	diphos	=dppe
BBN	borabicyclo[3.3.1]nonane	DIPT	diisopropyl tartrate
BCME	dis(chloromethyl)ether	DMA	dimethylacetamide
BHT	butylated hydroxytoluene (2,6-di- <i>t</i> -butyl- <i>p</i> -cresol)	DMAD	dimethyl acetylenedicarboxylate
BINAL-H	2,2'-dihydroxy-1,1'-binaphthyl-lithium aluminum hydride	DMAP	4-(dimethylamino)pyridine
BINAP	2,2'-bis(diphenylphosphino)-1,1'-binaphthyl	DME	1,2-dimethoxyethane
BINOL	1,1'-bi-2,2'-naphthol	DMF	dimethylformamide
bipy	2,2'-bipyridyl	dmg	dimethylglyoximate
BMS	borane-dimethyl sulfide	DMPU	<i>N,N'</i> -dimethylpropyleneurea
Bn	benzyl	DMS	dimethyl sulfide
Boc	<i>t</i> -butoxycarbonyl	DMSO	dimethyl sulfoxide
BOM	benzyloxymethyl	DMTSF	dimethyl(methylthio) sulfonium tetrafluoroborate
bp	boiling point	dppb	1,4-bis(diphenylphosphino)butane
Bs	brosyl (4-bromobenzenesulfonyl)	dppe	1,2-bis(diphenylphosphino)ethane
BSA	<i>N,O</i> -bis(trimethylsilyl)acetamide	dppf	1,1'-bis(diphenylphosphino)ferrocene
Bu	<i>n</i> -butyl	dppp	1,3-bis(diphenylphosphino)propane
Bz	benzoyl	DTBP	di- <i>t</i> -butyl peroxide
CAN	cerium(IV) ammonium nitrate	EDA	ethyl diazoacetate
Cbz	benzyloxycarbonyl	EDC	1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide
CDI	<i>N,N'</i> -carbonyldiimidazole	EDCI	= EDC
CHIRAPHOS	2,3-bis(diphenylphosphino)butane	ee	enantiomeric excess
Chx	= Cy	EE	1-ethoxyethyl
cod	cyclooctadiene	Et	ethyl
cot	cyclooctatetraene	ETSA	ethyl trimethylsilylacetate
Cp	cyclopentadienyl	EWG	electron withdrawing group
CRA	complex reducing agent	Fc	ferrocenyl
CSA	10-camphorsulfonic acid	Fmoc	9-fluorenylmethoxycarbonyl
CSI	chlorosulfonyl isocyanate	fp	flash point
Cy	cyclohexyl	Hex	<i>n</i> -hexyl
<i>d</i>	density	HMDS	hexamethyldisilazane
DABCO	1,4-diazabicyclo[2.2.2]octane	HMPA	hexamethylphosphoric triamide
DAST	<i>N,N'</i> -diethylaminosulfur trifluoride	HOBt	1-hydroxybenzotriazole
dba	dibenzylideneacetone	HOBt	=HOBt
DBAD	di- <i>t</i> -butyl azodicarboxylate	HOSu	<i>N</i> -hydroxysuccinimide
DBN	1,5-diazabicyclo[4.3.0]non-5-ene	Im	imidazole (imidazolyl)
DBU	1,8-diazabicyclo[5.4.0]undec-7-ene	Ipc	isopinocampheyl
DCC	<i>N,N'</i> -dicyclohexylcarbodiimide	IR	infrared
DCME	dichloromethyl methyl ether	KHDMS	potassium hexamethyldisilazide
DDO	dimethyldioxirane	LAH	lithium aluminum hydride
DDQ	2,3-dichloro-5,6-dicyano-1,4-benzoquinone	LD ₅₀	dose that is lethal to 50% of test subjects
de	diastereomeric excess		
DEAD	diethyl azodicarboxylate		
DET	diethyl tartrate		
DIBAL	diisobutylaluminum hydride		

LDA	lithium diisopropylamide	PMDTA	<i>N,N,N',N'',N'''</i> -pentamethyldiethylene-triamine
LDMAN	lithium 1-(dimethylamino)naphthalenide	PPA	polyphosphoric acid
LHMDS	= LiHMDS	PPE	polyphosphate ester
LICA	lithium isopropylcyclohexylamide	PPTS	pyridinium <i>p</i> -toluenesulfonate
LiHMDS	lithium hexamethyldisilazide	Pr	<i>n</i> -propyl
LiTMP	lithium 2,2,6,6-tetramethylpiperidine	PTC	phase transfer catalyst/catalysis
LTMP	= LiTMP	PTSA	<i>p</i> -toluenesulfonic acid
LTA	lead tetraacetate	py	pyridine
lut	lutidine		
<i>m</i> -CPBA	<i>m</i> -chloroperbenzoic acid	RAMP	(<i>R</i>)-1-amino-2-(methoxymethyl)pyrrolidine
MA	maleic anhydride	rt	room temperature
MAD	methylaluminum bis(2,6-di- <i>t</i> -butyl-4-methylphenoxide)	salen	bis(salicylidene)ethylenediamine
MAT	methylaluminum bis(2,4,6-tri- <i>t</i> -butylphenoxide)	SAMP	(<i>S</i>)-1-amino-2-(methoxymethyl)pyrrolidine
Me	methyl	SET	single electron transfer
MEK	methyl ethyl ketone	Sia	siamyl (3-methyl-2-butyl)
MEM	(2-methoxyethoxy)methyl	TASF	tris(diethylamino)sulfonium difluorotrimethylsilicate
MIC	methyl isocyanate	TBAB	tetrabutylammonium bromide
MMPP	magnesium monoperoxyphthalate	TBAF	tetrabutylammonium fluoride
MOM	methoxymethyl	TBAD	= DBAD
MoOPH	oxodiperoxomolybdenum(pyridine)-(hexamethylphosphoric triamide)	TBAI	tetrabutylammonium iodide
mp	melting point	TBAP	tetrabutylammonium perruthenate
MPM	= PMB	TBDMS	<i>t</i> -butyldimethylsilyl
Ms	mesyl (methanesulfonyl)	TBDPS	<i>t</i> -butyldiphenylsilyl
MS	mass spectrometry; molecular sieves	TBHP	<i>t</i> -butyl hydroperoxide
MTBE	methyl <i>t</i> -butyl ether	TBS	= TBDMS
MTM	methylthiomethyl	TCNE	tetracyanoethylene
MVK	methyl vinyl ketone	TCNQ	7,7,8,8-tetracyanoquinodimethane
<i>n</i>	refractive index	TEA	triethylamine
NaHDMS	sodium hexamethyldisilazide	TEBA	triethylbenzylammonium chloride
Naph	naphthyl	TEBAC	= TEBA
NBA	<i>N</i> -bromoacetamide	TEMPO	2,2,6,6-tetramethylpiperidinoxyl
nbd	norbornadiene (bicyclo[2.2.1]hepta-2,5-diene)	TES	triethylsilyl
NBS	<i>N</i> -bromosuccinimide	Tf	triflyl (trifluoromethanesulfonyl)
NCS	<i>N</i> -chlorosuccinimide	TFA	trifluoroacetic acid
NIS	<i>N</i> -iodosuccinimide	TFAA	trifluoroacetic anhydride
NMO	<i>N</i> -methylmorpholine <i>N</i> -oxide	THF	tetrahydrofuran
NMP	<i>N</i> -methyl-2-pyrrolidinone	THP	tetrahydropyran; tetrahydropyranyl
NMR	nuclear magnetic resonance	Thx	thexyl (2,3-dimethyl-2-butyl)
NORPHOS	bis(diphenylphosphino)bicyclo[2.2.1]-hept-5-ene	TIPS	triisopropylsilyl
Np	= Naph	TMANO	trimethylamine <i>N</i> -oxide
PCC	pyridinium chlorochromate	TMEDA	<i>N,N,N',N''</i> -tetramethylethylenediamine
PDC	pyridinium dichromate	TMG	1,1,3,3-tetramethylguanidine
Pent	<i>n</i> -pentyl	TMS	trimethylsilyl
Ph	phenyl	Tol	<i>p</i> -tolyl
phen	1,10-phenanthroline	TPAP	tetrapropylammonium perruthenate
Phth	phthaloyl	TBHP	<i>t</i> -butyl hydroperoxide
Piv	pivaloyl	TPP	tetraphenylporphyrin
PMB	<i>p</i> -methoxybenzyl	Tr	trityl (triphenylmethyl)
		Ts	tosyl (<i>p</i> -toluenesulfonyl)
		TTN	thallium(III) nitrate
		UHP	urea-hydrogen peroxide complex
		Z	= Cbz

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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 succeeded admirably in approaching 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 came to recognize that the large size of this eight-volume work and its cost of purchase often deterred 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 the same into a set of handbooks. The latter would also be purchasable on a single unit basis.

The ultimate result of these deliberations was the publication of the *Handbook of Reagents for Organic Synthesis*, the first four volumes of which appeared in 1999:

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

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

Edited by Robert M. Coates and Scott E. Denmark

Since then, the fifth and sixth members of this series have made their appearance in 2003 and 2005, respectively:

Chiral Reagents for Asymmetric Synthesis

Edited by Leo A. Paquette

Reagents for High-Throughput Solid-Phase and Solution-Phase Organic Synthesis

Edited by Peter Wipf

Each of the volumes contains a selected compilation of those entries from the original *Encyclopedia* that bear on the specific topic. The coverage of the last two handbooks also extends to the electronic sequel *e-EROS*. 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 present volume, entitled *Reagents for Glycoside, Nucleotide, and Peptide Synthesis*, constitutes the seventh entry in this continuing series of utilitarian reference works. As with its predecessors, this handbook is intended to be an affordable, enlightening compilation that will hopefully find its way into the laboratories of all practicing synthetic chemists. Every attempt has been made to be of the broadest possible relevance and the expectation is that our colleagues will share in this opinion.

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Introduction

With the advent of Chemical Biology, mainstream organic and medicinal chemists are increasingly aware of the need for, and are turning their attention to, the synthesis of bioconjugates. These might take the form of more classical peptides, nucleotides, or oligosaccharides, or some hybrid thereof, and may be required in straightforward soluble forms or appended to soluble or insoluble polymeric supports, nanoparticles, dendrimers, gold or silicon surfaces, or microtiter plates. Whatever the system or eventual use, the need for the efficient synthesis of glycoside, nucleotide, or peptide bonds has never been greater. The synthesis of each of these three main classes of biopolymers has evolved into a fine and sophisticated art in recent years with many reagents available to the experienced practitioner. The aim of this handbook is to collect the more important of these reagents together into a convenient, single volume and so aid the non-specialist in locating information and in making an informed choice when the need for the synthesis of a glycoside, nucleotide, or peptide bond arises. This handbook focuses on the chemical reagents required for glycoside, nucleotide, and peptide bond: information on polymeric supports and a broader selection of supported reagents, obviously very important considerations in this area, can be found in the recent *Handbook of Reagents for High-Throughput Solid-Phase and Solution-Phase Organic Synthesis* edited by Professor Peter Wipf.

Of the reagents featured in this volume, approximately one third are taken from the *Encyclopedia of Reagents for Organic Synthesis (EROS)*, published in 1995. Many of these are classical reagents in the field whose principal use has not changed in the intervening period. The remainder, and indeed the bulk, of the entries are comprised either of completely new articles, or of updated versions of original *EROS* articles taking into account recent developments, written by experts in the field for the continually-expanding online encyclopedia (*e-EROS*). The volume opens with a selection of recent monographs and review articles intended to help the reader in searching the literature. This list is divided into three sections: Glycoside, Nucleotide, and Peptide Bond Formation. The main sequence of reagents in this volume is alphabetical in keeping with the *EROS* and *e-EROS* format.

Finally, it is hoped that this volume will be a useful and convenient handbook for any researcher involved in the synthesis of bioconjugates, perhaps even helping to identify present shortcomings in the field and, so, spur the design and development of even more and better reagents for the same.

David Crich

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