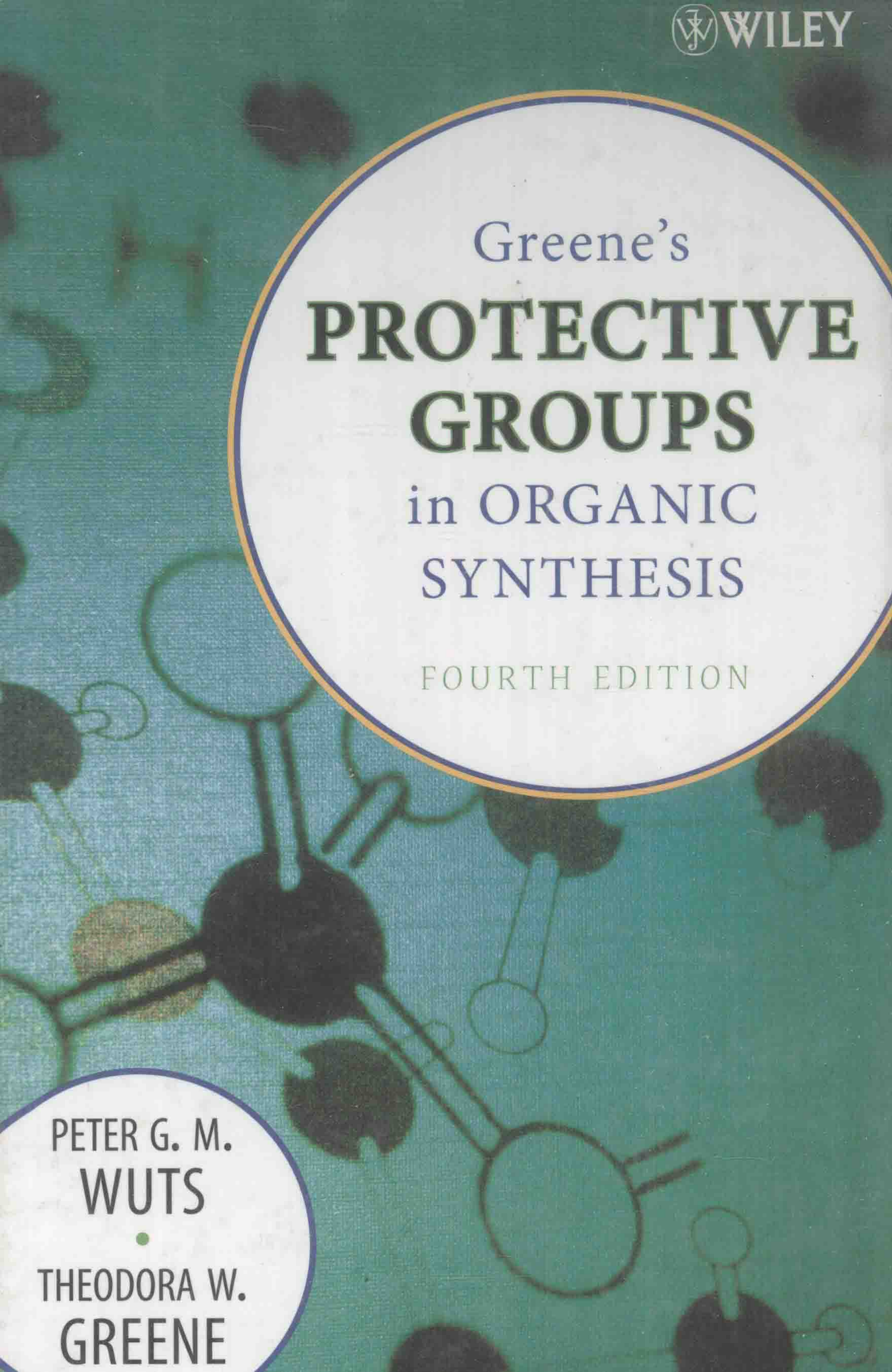

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Greene's

PROTECTIVE GROUPS in ORGANIC SYNTHESIS

FOURTH EDITION

PETER G. M.
WUTS

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THEODORA W.
GREENE

GREENE'S PROTECTIVE GROUPS IN ORGANIC SYNTHESIS

Fourth Edition

PETER G. M. WUTS
Pfizer

and

THEODORA W. GREENE
The Rowland Institute for Science

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PREFACE TO THE FOURTH EDITION

After completing the mammoth third edition, I never imagined that a fourth edition would eventuate because of the sheer volume of literature that must be examined to cover the subject comprehensively. Nonetheless, I took on the task with the encouragement and help of my wife, Lizzie, who agreed to assist me with this one, since Theo was not able to. As with the last edition, the searches were primarily done by hand because databases such as Scifinder fail to be selective and have such a prodigious output that no one can be expected to filter all that material in a reasonable amount of time. Nevertheless, Scifinder was used to locate material in journals which were not readily accessible. In recent years, in both corporate and academic America, there has also been a trend to do away with physical libraries, which makes doing a literature search extremely difficult, especially if you like reading the literature at home in a comfortable chair. Reading journals on a computer screen may be easy for Spock, but I find it difficult and stressful. With limited access to hard copies of some of the literature, I may have missed some things. For this I apologize and will not be offended if the author sends me the material for inclusion in a possible future edition. The literature search is complete through the end of 2005.

With that said, the fourth edition contains over 3100 new references compared to the 2349 new citations in the third edition. In keeping with the tradition of the past, I tried to include material covering new methods for existing protective groups along with new groups that have been developed. When the authors disclosed the information, I also provided the rationale for the choice of a given protective group. In that synthetic chemistry is still not sufficiently developed to do away with protective groups altogether, I have included many examples that highlight selective protection and deprotection, especially when the selectivity might not be totally obvious or expected. Issues of unexpected reactivity are also included, since these cases should help in choosing a group during the development of a synthetic plan. On the whole, this is a book of options for the synthetic chemist, since no one method is suitable for all occasions. Also, many of the published methods have not been tested in complex situations; thus it is impossible to determine which method of a particular set might be the best, and, as such, no attempt was made to try and order the various methods that appear in a section. The issue of functional group compatibility is often not addressed in papers describing new methods, and this further complicates the evaluation process. Comparative studies for either protection or deprotection are rarely done and as a result, trial and error and chemical intuition must be used to define the most suitable method in a given situation.

All sections of the book have seen some expansion, especially the chapters on alcohol and amine protection. I had considered adding a section that covered areas such as diene protection as metal complexes and Diels–Alder adducts, but the use of these is rather limited. The Reactivity Charts of Chapter 10 have not been altered, but a new chart covering selectivity in silyl group deprotection has been added. The overall format of the book has been retained and in some of the larger sections, similar methods have been grouped together. A new area has emerged since the last edition, and this is the use of fluorous protective groups. These have been included and placed in the appropriate sections rather than having collected them together.

The completion of this project was aided by a number of people. First of all this work would not have been started without the encouragement and dedication of my wife, Lizzie, who looked up and downloaded many of the references and then typed every new reference into an Endnote™ database. She double-checked the entire set in order to prevent errors. She also read through the entire manuscript to check it for punctuation, grammar, and consistency. She has a degree in Near Eastern Medieval History, thus I take full responsibility for any chemical errors. I must also thank her for not complaining about becoming a book widow while I spent countless hours on this project over a period of ~3 years. A special note of thanks must be extended to Peter Green, the Pfizer Michigan site head, who approved giving Lizzie access to the company library system even though she was not an employee. I would also like to thank Jake Szmuszkovicz, Raymond Conrow, and Martin Lang for providing me with references to be included in the fourth edition, and finally I wish to thank Joseph Muchowski for bringing an error in the third edition, now corrected, to my attention.

PETER G. M. WUTS

July 2006

PREFACE TO THE THIRD EDITION

Organic synthesis has not yet matured to the point where protective groups are not needed for the synthesis of natural and unnatural products; thus, the development of new methods for functional group protection and deprotection continues. The new methods added to this edition come from both electronic searches and a manual examination of all the primary journals through the end of 1997. We have found that electronic searches of *Chemical Abstracts* fail to find many new methods that are developed during the course of a synthesis, and issues of selectivity are often not addressed. As with the second edition, we have attempted to highlight unusual and potentially useful examples of selectivity for both protection and deprotection. In some areas the methods listed may seem rather redundant, such as the numerous methods for THP protection and deprotection, but we have included them in an effort to be exhaustive in coverage. For comparison, the first edition of this book contains about 1500 references and 500 protective groups, the second edition introduces an additional 1500 references and 206 new protective groups, and the third edition adds 2349 new citations and 348 new protective groups.

Two new sections on the protection of phosphates and the alkyne-CH are included. All other sections of the book have been expanded, some more than others. The section on the protection of alcohols has increased substantially, reflecting the trend of the nineties to synthesize acetate- and propionate-derived natural products. An effort was made to include many more enzymatic methods of protection and deprotection. Most of these are associated with the protection of alcohols as esters and the protection of carboxylic acids. Here we have not attempted to be exhaustive, but hopefully, a sufficient number of cases are provided that illustrate the true power of this technology, so that the reader will examine some of the excellent monographs and review articles cited in the references. The Reactivity Charts in Chapter 10 are identical to those in the first edition. The chart number appears beside the name of each protective group when it is first introduced. No attempt was made to update these Charts, not only because of the sheer magnitude of the task, but because it is nearly impossible in a two-dimensional table to address adequately the effect that electronic and steric controlling elements have on a particular instance of protection or deprotection. The concept of fuzzy sets as outlined by Lofti Zadeh would be ideally suited for such a task.

The completion of this project was aided by the contributions of a number of people. I am grateful to Rein Virkhaus and Gary Callen, who for many years forwarded me references when they found them, to Jed Fisher for the information he contributed on phosphate protection, and to Todd Nelson for providing me a preprint of his excellent review article on the deprotection of silyl ethers. I heartily thank Theo Greene for checking and rechecking the manuscript—all 15 cm of it—for spelling and consistency and for the arduous task of checking all the references for accuracy. I thank Fred Greene for reading the manuscript, for his contribution to Chapter 1 on the use of protective groups in the synthesis of himastatin, and for his contribution to the introduction to Chapter 9, on phosphates. I thank my wife, Lizzie, for encouraging me to undertake the third edition, for the hours she spent in the library looking up and photocopying hundreds of references, and for her understanding while I sat in front of the computer night after night and numerous weekends over a two-year period. She is the greatest!

PETER G. M. WUTS

Kalamazoo, Michigan
June 1998

PREFACE TO THE SECOND EDITION

Since publication of the first edition of this book in 1981, many new protective groups and many new methods of introduction or removal of known protective groups have been developed: 206 new groups and approximately 1500 new references have been added. Most of the information from the first edition has been retained. To conserve space, generic structures used to describe Formation/Cleavage reactions have been replaced by a single line of conditions, sometimes with explanatory comments, especially about selectivity. Some of the new information has been obtained from on-line searches of *Chemical Abstracts*, which have limitations. For example, *Chemical Abstracts* indexes a review article about protective groups only if that word appears in the title of the article. References are complete through 1989. Some references, from more widely circulating journals, are included for 1990.

Two new sections on the protection for indoles, imidazoles, and pyrroles and protection for the amide –NH are included. They are separated from the regular amines because their chemical properties are sufficiently different to affect the chemistry of protection and deprotection. The Reactivity Charts in Chapter 8 are identical to those in the first edition. The chart number appears beside the name of each protective group when it is first discussed.

A number of people must be thanked for their contributions and help in completing this project. I am grateful to Gordon Bundy, who loaned me his card file, which provided many references that the computer failed to find, and to Bob Williams, Spencer Knapp, and Tohru Fukuyama for many references on amine and amide protection. I thank Theo Greene who checked and rechecked the manuscript for spelling and consistency and for the herculean task of checking all the references to make sure that my 3's and 8's and 7's and 9's were not interchanged—all done without a single complaint. I thank Fred Greene who read the manuscript and provided valuable suggestions for its improvement. My wife Lizzie was a major contributor to getting this project finished, by looking up and photocopying references, by turning on the computer in an evening ritual, and by typing many sections of the original book, which made the changes and additions much easier. Without her understanding and encouragement, the volume probably would never have been completed.

PETER G. M. WUTS

Kalamazoo, Michigan
May 1990

PREFACE TO THE FIRST EDITION

The selection of a protective group is an important step in synthetic methodology, and reports of new protective groups appear regularly. This book presents information on the synthetically useful protective groups (~500) for five major functional groups: $-OH$, $-NH$, $-SH$, $-COOH$, and $>C{=}O$. References through 1979, the best method(s) of formation and cleavage, and some information on the scope and limitations of each protective group are given. The protective groups that are used most frequently and that should be considered first are listed in Reactivity Charts, which give an indication of the reactivity of a protected functionality to 108 prototype reagents.

The first chapter discusses some aspects of protective group chemistry: the properties of a protective group, the development of new protective groups, how to select a protective group from those described in this book, and an illustrative example of the use of protective groups in a synthesis of brefeldin. The book is organized by functional group to be protected. At the beginning of each chapter are listed the possible protective groups. Within each chapter protective groups are arranged in order of increasing complexity of structure (e.g., methyl, ethyl, *t*-butyl, . . . , benzyl). The most efficient methods of formation or cleavage are described first. Emphasis has been placed on providing recent references, since the original method may have been improved. Consequently, the original reference may not be cited; my apologies to those whose contributions are not acknowledged. Chapter 8 explains the relationship between reactivities, reagents, and the Reactivity Charts that have been prepared for each class of protective groups.

This work has been carried out in association with Professor Elias J. Corey, who suggested the study of protective groups for use in computer-assisted synthetic analysis. I appreciate his continued help and encouragement. I am grateful to Dr. J. F. W.

McOmie (Ed., *Protective Groups in Organic Chemistry,* Plenum Press, New York and London, 1973) for his interest in the project and for several exchanges of correspondence, and to Mrs. Mary Fieser, Professor Frederick D. Greene, and Professor James A. Moore for reading the manuscript. Special thanks are also due to Halina and Piotr Starewicz for drawing the structures, and to Kim Chen, Ruth Emery, Janice Smith, and Ann Wicker for typing the manuscript.

THEODORA W. GREENE

Harvard University
September 1980

ABBREVIATIONS

PROTECTIVE GROUPS

In some cases, several abbreviations are used for the same protective group. We have listed the abbreviations as used by an author in his original paper, including capital and lowercase letters. Occasionally, the same abbreviation has been used for two different protective groups. This information is also included.

ABO	2,7,8-trioxabicyclo[3.2.1]octyl
Ac	acetyl
ACBZ	4-azidobenzyloxycarbonyl
ACE	*O*-bis(2-Acetoxyethoxy)methyl
AcHmb	2-acetoxy-4-methoxybenzyl
Acm	acetamidomethyl
Ad	1-adamantyl
ADMB	4-acetoxy-2,2-dimethylbutanoate
Adoc	1-adamantyloxycarbonyl
Adpoc	1-(1-adamantyl)-1-methylethoxycarbonyl
Alloc or AOC	allyloxycarbonyl
AOC or Alloc	allyloxycarbonyl
Allocam	allyloxycarbonylaminomethyl
Als	allylsulfonyl
AMB	2-(acetoxymethyl)benzoyl
AMPA	(2-azidomethyl)phenylacetate
AN	4-methoxyphenyl or anisyl
Ans	anisylsulfonyl

Anpe	2-(4-acetyl-2-nitrophenyl)ethyl
p-AOM	*p*-anisyloxymethyl or (4-methoxyphenoxy)methyl
APAC	2-allyloxyphenylacetate
Aqmoc	anthraquinone-2-ylmethoxycarbonyl
Azb	*p*-azidobenzyl
Azm	azidomethyl
AZMB	2-(azidomethyl)benzoate
Bam	benzamidomethyl
BBA	butane-2,3-bisacetal
Bbc	but-2-ynylbisoxycaronyl
BDIPS	biphenyldiisopropylsilyl
BDMS	biphenyldimethylsilyl benzyldimethylsilyl
Bdt	1,3-benzodithiolan-2-yl
Betsyl or Bts	benzothiazole-2-sulfonyl
Bhcmoc	6-bromo-7-hydroxycoumarin-4-ylmethoxycarbonyl
BHQ	8-bromo-7-hydroxyquinoline-2-ylmethyl
BHT	2,6-di-*t*-butyl-4-methylphenyl
Bic	5-benzisoxazolylmethoxycarbonyl
Bim	5-benzisoazolylmethylene
Bimoc	benz[*f*]inden-3-ylmethoxycarbonyl
BIPSOP	*N*-2,5-bis(triisopropylsiloxy)pyrrolyl
BMB	*o*-(benzoyloxymethyl)benzoyl
Bmpc	2,4-dimethylthiophenoxycarbonyl
Bmpm	bis(4-methoxyphenyl)-1′-pyrenylmethyl
Bn	benzyl
Bnf	fluorousbenzyl
Bnpeoc	2,2-bis(4′-nitrophenyl)ethoxycarbonyl
Bns	benzylsulfonate
BOB	benzyloxybutyrate
BOC	*t*-butoxycarbonyl
Bocdene	2-(*t*-butylcarbonyl)ethylidene
BOM	benzyloxymethyl
Bpoc	1-methyl-1-(4-biphenyl)ethoxycarbonyl
BSB	benzoSTABASE
Bsmoc	1,1-dioxobenzo[*b*]thiophene-2-ylmethoxycarbonyl
BTM	*t*-butylthiomethyl
Bts or Betsyl	benzothiazole-2-sulfonyl
B^{t}SE	2-*t*-butylsulfonylethyl
Bts-Fmoc	2,7-bis(trimethylsilyl)fluorenylmethoxycarbonyl
Bum	*t*-butoxymethyl
Bus	*t*-butylsulfonyl
t-Bumeoc	1-(3,5-di-*t*-butylphenyl)-1-methylethoxycarbonyl
Bz	benzoyl
CAEB	2-[(2-chloroacetoxy)ethyl]benzoyl

Cam	carboxamidomethyl
CAMB	2-(chloroacetoxymethyl)benzoyl
Cbz or Z	benzyloxycarbonyl
CEM	2-cyanoethoxymethyl
CDA	cyclohexane-1,2-diacetal
CDM	2-cyano-1,1-dimethylethyl
CE or Cne	2-cyanoethyl
Cee	1-(2-chloroethoxy)ethyl
CEE	1-(2-cyanoethoxy)ethyl
Ceof	cyclic ethyl orthoformate
cHex	cyclohexyl
Chx	cyclohexyl
Cin	cinnamyl
ClAzab	4-azido-3-chlorobenzyl
Climoc	2-chloro-3-indenylmethoxycarbonyl
Cms	carboxymethylsulfenyl
CNAP	2-naphthylmethoxycarbonyl
Cne or CE	2-cyanoethyl
Coc	cinnamyloxycarbonyl
CPC	*p*-chlorophenylcarbonyl
CPDMS	(3-cyanopropyl)dimethylsilyl
Cpeoc	2-(cyano-1-phenyl)ethoxycarbonyl
Cpep	1-(4-chlorophenyl)-4-methoxypiperidin-4-yl
CPTr	4,4′,4″-tris(4,5-dichlorophthalimido)-triphenylmethyl
CTFB	4-trifluoromethylbenzyloxycarbonyl
CTMP	1-[(2-chloro-4-methyl)phenyl]-4-methoxypiperidin-4-yl
Cyclo-SEM	5-trimethylsilyl-1,3-dioxane
Cys	cysteine
DAM	di-*p*-anisylmethyl or bis(4-methoxyphenyl)methyl
DATE	1,1-di-*p*-anisyl-2,2,2-trichloroethyl
DB-*t*-BOC	1,1-dimethyl-2,2-dibromoethoxycarbonyl
DBD-Tmoc	2,7-di-*t*-butyl[9-(10,10-dioxo-10,10,10,10-tetrahydrothioxanthyl)]methoxycarbonyl
DBS	dibenzosuberyl
DCP	dichlorophthalimide
Dcpm	dicyclopropylmethyl
Ddm or Dmbh	bis(4-methoxyphenyl)methyl
Dde	2-(4,4-dimethyl-2,6-dioxocyclohexylidene)ethyl
Ddz	1-methyl-1-(3,5-dimethoxyphenyl)ethoxycarbonyl
DEM	diethoxymethyl
DEIPS	diethylisopropylsilyl
Desyl	2-oxo-1,2-diphenylethyl
Dim	1,3-dithianyl-2-methyl

Dmab	4-{*N*-[1-(4,4-dimethyl-2,6-dioxocyclohexylidene)-3-methylbutyl]amino}benzyl
DMB	"3′,5′-dimethoxybenzoin"
Dmb	2,4-dimethoxybenzyl
DMBM	[(3,4-dimethoxybenzyl)oxy]methyl
DMIPS	dimethylisopropylsilyl
DMN	2,3-dimethylmaleimide
Dmoc	dithianylmethoxycarbonyl
Dmp	2,4-dimethyl-3-pentyl
Dmp	dimethylphosphinyl
DMP	dimethoxyphenyl dimethylphenacyl
DMPM	3,4-dimethoxybenzyl
DMTC	dimethylthiocarbamate
DMT or DMTr	di(*p*-methoxyphenyl)phenylmethyl or dimethoxytrityl
DMTr or DMT	di(*p*-methoxyphenyl)phenylmethyl or dimethoxytrityl
DNAP	2-(dimethylamino)-5-nitrophenyl
DNB	*p,p*′-dinitrobenzhydryl
DNMBS	4-(4′,8′-dimethoxynaphthylmethyl)benzenesulfonyl
DNP	2,4-dinitrophenyl
Dnpe	2-(2,4-dinitrophenyl)ethyl
Dnpeoc	2-(2,4-dinitrophenyl)ethoxycarbonyl
DNs	2,4-dinitrobenzenesulfonyl
DNse	2-(2,4-dinitrophenylsulfonyl)ethoxycarbonyl
Dnseoc	2-dansylethoxycarbonyl
Dobz	*p*-(dihydroxyboryl)benzyloxycarbonyl
Doc	2,4-dimethylpent-3-yloxycarbonyl
Dod	bis(4-methoxylphenyl)methyl
DOPS	dimethyl[1,1-dimethyl-3-(tetrahydro-2*H*-pyran-2-yloxy)propyl]silyl
DPA	diphenylacetyl
DPIPS	diphenylisopropylsilyl
DPM or Dpm	diphenylmethyl
DPMS	diphenylmethylsilyl
Dpp	diphenylphosphinyl
Dppe	2-(diphenylphosphino)ethyl
Dppm	(diphenyl-4-pyridyl)methyl
DPSE	2-(methyldiphenylsilyl)ethyl
DPSide	diphenylsilyldiethylene
Dpt	diphenylphosphinothioyl
DPTBOS	*t*-Butoxydiphenylsilyl
DPTBS	diphenyl-*t*-butoxysilyl or diphenyl-*t*-butylsilyl
Dtb-Fmoc	2,6-di-*t*-butyl-9-fluorenylmethoxycarbonyl
DTBMS	di-*t*-butylmethylsilyl

DTBS	di-*t*-butylsilylene
DTE	2-(hydroxyethyl)dithioethyl or “dithiodiethanol”
Dts	dithiasuccinimidyl
E-DMT	1,2-ethylene-3,3-bis(4′4″-dimethoxytrityl)
EE	1-ethoxyethyl
EOM	ethoxymethyl
^{F}Cbz	fluorous benzyloxycarbonyl
Fcm	ferrocenylmethyl
Flu	fluorenyl
Fm	9-fluorenylmethyl
Fmoc	9-fluorenylmethoxycarbonyl
Fpmp	1-(2-fluorophenyl)-4-methoxypiperidiny-4-yl
GUM	guaiacolmethyl
HAPE	1-[2-(2-hydroxyalkyl)phenyl]ethanone
HBn	2-hydroxybenzyl
Hdoc	hexadienyloxycarbonyl
HFB	hexafluoro-2-butyl
HIP	1,1,1,3,3,3-hexafluoro-2-phenylisopropyl
Hoc	cyclohexyloxycarbonyl
HSDIS	(hydroxystyryl)diisopropylsilyl
HSDMS	(hydroxystyryl)dimethylsilyl
hZ or homo Z	homobenzyloxycarbonyl
IDTr	3-(imidazol-1-ylmethyl)-4′,4″-dimethoxytriphenylmethyl
IETr	4,4′-dimethoxy-3″-[*N*-(imidazolylethyl) carbamoyl]trityl
iMds	2,6-dimethoxy-4-methylbenzenesulfonyl
Ipaoc	1-isopropylallyloxycarbonyl
Ipc	isopinocampheyl
IPDMS	isopropyldimethylsilyl
Lev	levulinoyl
LevS	4,4-(ethylenedithio)pentanoyl
LevS	levulinoyldithioacetal ester
LMM*o*(*p*)NBz	6-(levulinyloxymethyl)-3-methoxy-2-nitrobenzoate
MAB	2-{{[(4-methoxytrityl)thio]methylamino} methyl}benzoate
MAQ	2-(9,10-anthraquinonyl)methyl or 2-methyleneanthraquinone
MBE	1-methyl-1-benzyloxyethyl
Mbh	bis(4-methylphenyl)methyl
MBF	2,3,3a,4,5,6,7,7a-octahydro-7,8,8-trimethyl-4,7-methanobenzofuran-2-yl
MBS or Mbs	*p*-methoxybenzenesulfonyl
MCPM	1-methyl-1′-cyclopropylmethyl
Mds	2,6-dimethyl-4-methoxybenzenesulfonyl

MDPS	methylene-bis-(diisopropylsilanoxanylidene
Me	methyl
ME	methoxyethyl
MEC	α-methylcinnamyl
Mee	methoxyethoxyethyl
MeOAc	methoxyacetate
MEM	2-methoxyethoxymethyl
Menpoc	α-methylnitropiperonyloxycarbonyl
MeOZ or Moz	*p*-methoxybenzyloxycarbonyl
Mes	mesityl or 2,4,6-trimethylphenyl
MIP	methoxyisopropyl or 1-methyl-1-methoxyethyl
MM	menthoxymethyl
MMT or MMTr	*p*-methoxyphenyldiphenylmethyl
MMTr or MMT	*p*-methoxyphenyldiphenylmethyl
MMPPOC	2-(3,4-methylenedioxy-6-nitrophenypropyloxycarbonyl
MOB	2-{[(4-methoxytritylthio)oxy]methyl}benzoate
Mocdene	2-(methoxycarbonyl)ethylidene
MoEt	2-*N*-(morpholino)ethyl
MOM	methoxymethyl
MOMO	methoxymethoxy
Moz or MeOZ	*p*-methoxybenzyloxycarbonyl
MP	*p*-methoxyphenyl
Mpe	3-methyl-3-pentyl
MPM or PMB	*p*-methoxyphenylmethyl or *p*-methoxybenzyl
Mps	*p*-methoxyphenylsulfonyl
Mpt	dimethylphosphinothioyl
Ms	methanesulfonyl or mesyl
MSE	2-(methylsulfonyl)ethyl
Msib	4-(methylsulfinyl)benzyl
Mspoc	2-methylsulfonyl-3-phenyl-1-prop-2-enyloxy
Msz	4-methylsulfinylbenzyloxycarbonyl
MTAD	4-methyl-1,2,4-triazoline-3,5-dione
Mtb	2,4,6-trimethoxybenzenesulfonyl
Mte	2,3,5,6-tetramethyl-4-methoxybenzenesulfonyl
MTHP	4-methoxytetrahydropyranyl
MTM	methylthiomethyl
MTMB	4-(methylthiomethoxy)butyryl
MTMECO	2-(methylthiomethoxy)ethoxycarbonyl
MTMT	2-(methylthiomethoxymethyl)benzoyl
Mtpc	4-(methylthio)phenoxycarbonyl
Mtr	2,3,6-trimethyl-4-methoxybenzenesulfonyl
Mts	2,4,6-trimethylbenzenesulfonyl or mesitylenesulfonyl
Mtt	4-methoxytrityl or 4-methyltrityl
Nap	2-napthylmethyl