2007
VOLUME 7
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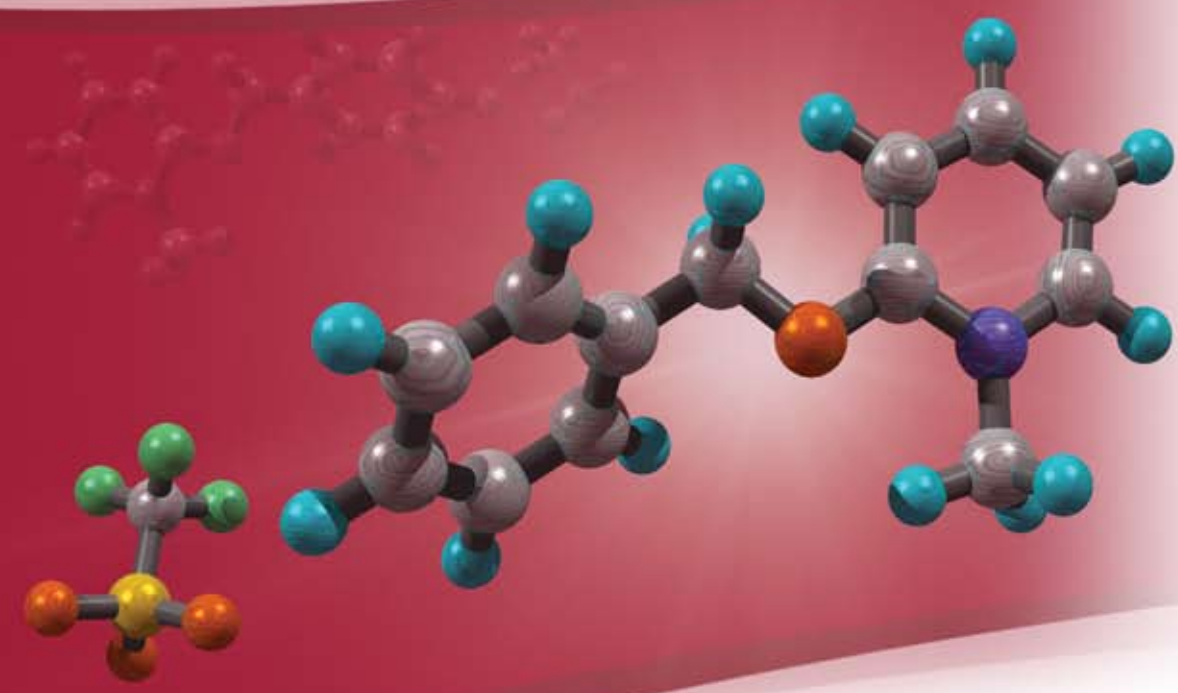
Protection



2-Benzyloxy-1-methylpyridinium triflate: an air-stable pre-activated pyridinium salt for the mild benzylation of alcohols under neutral conditions.

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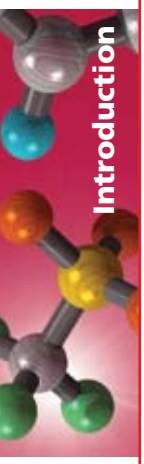

Introduction

One of the common difficulties with natural product and other multi-step syntheses is the need to render one functional group inert to a particular reagent while keeping another group open for further chemical elaboration. Despite the great advances made in the involved syntheses of multifunctional products, selectivity in functional group transformations remains a critical issue in organic synthesis. Unfortunately for the synthetic chemist, there is no perfect protecting group applicable to any functional group in any situation. Thus, the need exists for the synthetic chemist to have a handy toolbox of selective and efficient protecting groups that can be applied and easily removed under a variety of conditions.

In this issue, we are pleased to introduce a few recent additions to the available protection reagents we offer. The Dudley Reagent is capable of benzylation of alcohols under neutral conditions. Allyl and 4-methoxybenzyl trichloroacetimidates are also commonly used to protect alcohols in various syntheses. Ethynylnaphthalenes offer sterically unobtrusive protection of hydroxyl groups on carbohydrates with orthogonal reactivity compared to benzyl ethers. The (2-trimethylsilyl)ethanesulfonyl (SES) group is used to protect amines via SES chloride; alternatively, SES-NH_2 can be used to introduce a SES-protected amine functionality directly into a molecule.

We also would like to introduce a variety of fluorous protecting groups to our protection line. Fluorous protecting groups serve dual purposes – they are able to both act as a protecting group as well as serve as a temporary fluorous tag that can facilitate product workup and purification throughout the synthesis. Finally, we have included the most popular reagents we offer for the protection of alcohols and amines.

For a complete listing of protection reagents, please visit **sigma-aldrich.com/protection**. If you are unable to find the specific reagent for your research, "Please Bother Us" with your suggestions at **mredlich@sial.com**, or contact your local Sigma-Aldrich office (see back cover).

About Our Cover

The cover graphic depicts the structure of the Dudley Benzylation Reagent, 2-benzyloxy-1-methylpyridinium triflate. This bench-stable, mild benzylation reagent protects alcohols under neutral conditions, and succeeds in cases where the benzyl trichloroacetimidate does not yield the desired product. Simply heating the alcohol in the presence of the salt provides the desired benzyl ether, and byproducts are easily removed from the reaction mixture.

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Dudley Benzylation Reagent

Benzyl ethers and derivatives are among the most widely used protecting groups in organic synthesis. Cleavage can be effected under a variety of conditions including hydrogenolysis, oxidation, and acid decomposition. Typically, protection of alcohols in the form of a benzyl ether requires harsh reaction conditions. Williamson ether synthesis necessitates strongly basic conditions to generate an alkoxide nucleophile (**Figure 1**). Alternatively, trichloroacetimidate reagents can be employed in the presence of triflic acid (HOTf) as a promoter. Many complex alcohols are incompatible with these strongly basic or acidic conditions. For example, β-hydroxy esters are prone to elimination, epimerization at the α-carbon, or retro-aldol reactions under acid or base catalysis. Additionally, resident protecting groups on the alcohol substrate may be incompatible with non-pH-neutral reactions. Under acidic conditions, trimethylsilyl (TMS) ethers are easily cleaved, while acetals can undergo migration in polyol systems. Bulkier silyl protecting groups can undergo migration in the presence of base.

Professor Gregory Dudley and co-workers at Florida State University have developed a pre-activated pyridinium salt for the mild benzylation of alcohols under neutral conditions.[1] The salt is bench-stable, can be handled in air (**Figure 2**), and simple reaction conditions are employed. Simply heating the alcohol in the presence of the salt provides the desired benzyl ether. Heterogeneous MgO serves to neutralize the mildly acidic hydroxypyridine generated during the protection reaction. The resultant pyridinone byproduct is water soluble, and thus, easily removed (**Scheme 1**).

As shown in **Table 1**, a variety of primary, secondary, and tertiary alcohols underwent clean and high-yielding benzylation. 1,2-Dichloroethane (DCE), benzene, toluene, and benzotrifluoride (BTF) are viable solvents for the reaction. BTF is a low-cost, moderately volatile solvent that is an environmentally friendly alternative to chlorinated solvents. Notably, the labile stereogenic center of optically pure methyl 3-hydroxy-2-methylpropionate survived the reaction conditions unaltered (entry 6). Trimethylsilylethanol (entry 7) is subject to Peterson elimination under acidic or basic conditions, and its benzyl ether had not been reported previously. Attempts to generate the benzyl ether derivative with benzyl trichloroacetimidate did not provide any of the desired product. However, use of the pyridinium reagent cleanly provided the alcohol in 100% conversion.

References: (1) (a) Poon, K. W. C.; Dudley, G. B. *J. Org. Chem.* **2006**, *71*, 3923. (b) Poon, K. W. C. et al. *Synlett* **2005**, 3142.

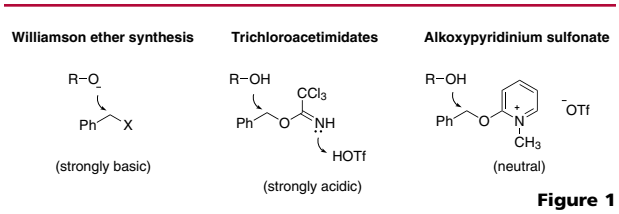
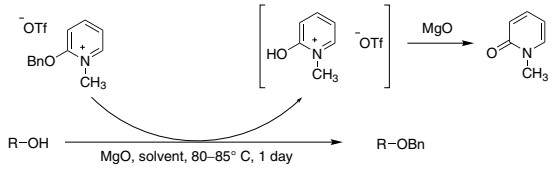
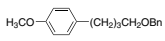
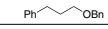
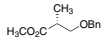
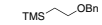
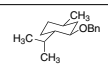


Figure 1

Figure 2



Scheme 1

Entry	Benzylation Product	Solvent	^{1}H NMR Yield (%)
1		DCE	67
2		benzene	93
3		toluene	91
4		BTF	>95
5		BTF	>95
6		BTF	85
7		BTF	100*
8		BTF	88
9		BTF	80

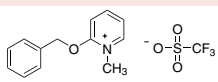
*conversion

Table 1

2-Benzyloxy-1-methylpyridinium triflate

NEW

$C_{14}H_{14}F_3NO_4S$
FW: 349.33
[*26189-59-3*]



679674-1G	1 g
679674-5G	5 g

Trichloroacetimidate Reagents

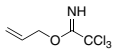
As described previously, trichloroacetimidates are also commonly employed as alcohol alkylation reagents, particularly when existing functionality is not acid sensitive.[1] Recent applications of allyl trichloroacetimidate include the synthesis of an allyl propargyl ether intermediate in the synthesis of the fused-ring alkaloid securinine,[2] preparation of fluorinated probes for protein kinase C (PKC),[3] and in the formal synthesis of the oxocene target Laurencin (**Scheme 1**).[4]

Likewise, 4-methoxybenzyl trichloroacetimidate has found extensive application for the protection of alcohols in the form of *p*-methoxybenzyl (PMB) ethers that are readily cleaved under oxidative conditions, typically dichlorodicyanoquinone (DDQ) or ceric ammonium nitrate (CAN). As shown in **Scheme 2**, 4-methoxybenzyl trichloroacetimidate was successfully applied in the selective preparation of chiral *syn*- or *anti*- diamines,[5] as well as in independent syntheses of bryostatin intermediates (**Scheme 3**).[6,7]

References: (1) Greene, T. W.; Wuts, P. G. M. *Protective Groups in Organic Synthesis*, 3rd ed.; John Wiley & Sons: New York, 1999. (2) Honda, T. et al. *Org. Lett.* **2004**, *6*, 87. Attempted allylation with allyl bromide under basic conditions did not provide the desired compound. (3) Goekjian, P. G. et al. *J. Org. Chem.* **1999**, *64*, 4238. (4) Krüger, J.; Hoffmann, R. W. *J. Am. Chem. Soc.* **1997**, *119*, 7499. Attempted allylation under basic conditions resulted in silyl migration. (5) Ichikawa, Y. et al. *Org. Lett.* **2006**, *8*, 5737. (6) Manaviazar, S. et al. *Org. Lett.* **2006**, *8*, 4477. (7) Keck, G. E. et al. *Org. Lett.* **2006**, *8*, 3667.

O-Allyl 2,2,2-trichloroacetimidate, 96% NEW

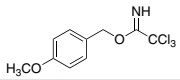
$C_5H_6Cl_3NO$
FW: 202.47
[*51479-73-3*]



678414-5G	5 g

4-Methoxybenzyl-2,2,2-trichloroacetimidate NEW

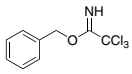
$C_{10}H_{10}Cl_3NO_2$
FW: 282.55
[*89238-99-3*]



679585-5G	5 g
679585-25G	25 g

Benzyl 2,2,2-trichloroacetimidate, 99%

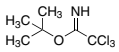
$C_9H_8Cl_3NO$
FW: 252.52
[*81927-55-1*]



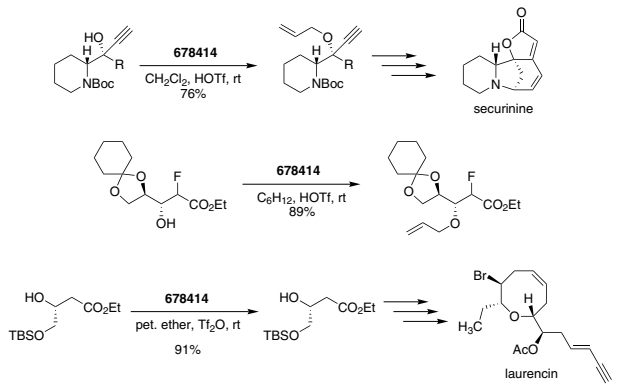
140333-5G	5 g
140333-25G	25 g

tert-Butyl 2,2,2-trichloroacetimidate, 96%

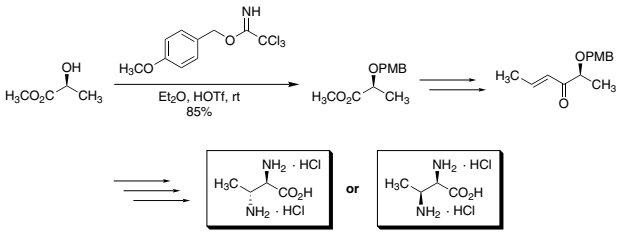
$C_6H_{10}Cl_3NO$
FW: 218.51
[*98946-18-0*]



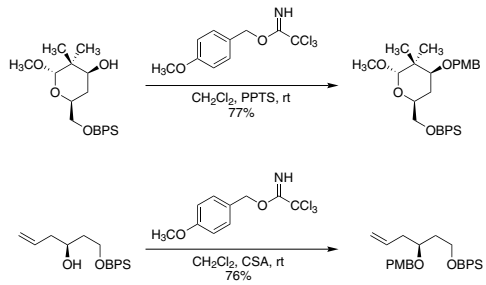
364789-1G	1 g
364789-5G	5 g
364789-25G	25 g



Scheme 1



Scheme 2



Scheme 3

(2-Trimethylsilyl)-ethanesulfonyl Reagents

In 1986, Weinreb first reported the (2-trimethylsilyl)ethanesulfonyl (SES) group (**Figure 1**) as an alternative to a tosyl sulfonamide for mild sulfonyl protection of amines.[1] The SES-protected amines are stable compounds that can be readily cleaved by fluoride sources to regenerate the parent free amine and other volatile products, whereas the tosyl sulfonamide often proves difficult to deprotect.[2]

SES-Cl can be used to protect amino acids, and can be carried through syntheses as easily as Boc-, Fmoc-, or Z-protected amino acids. Boger and co-workers have recently employed SES-Cl to protect the amino side chain on D- or L-ornithine prior to protection of the carboxylic acid function (**Scheme 1**). Boger used these protected ornithines in their syntheses of Ramoplanin analogues[3] and the cyclic peptide of Chlorofusin.[4]

Ohno and Tanaka also used the SES protecting group in the palladium(0)-catalyzed synthesis of 1,4-oxazepines. The facile deprotection of the SES group compared to the tosyl group allowed for the creation of the heterocycle product bearing a free amino group (**Scheme 2**).[5]

In certain reactions, 2-(trimethylsilyl)ethanesulfonamide (SES-NH_2), can be used to directly introduce a protected nitrogen-functionality into a substrate. One example is in the work of Bolm and Mancheño, who reported the use of SES-NH_2 in the iron-catalyzed imination of sulfoxides to yield sulfoximines (**Scheme 3**).[6]

Lamaty and co-workers have employed SES-NH_2 in an *aza*-Baylis–Hillman reaction to prepare a series of SES-protected β-aminoesters. These β-aminoesters were then further elaborated to provide a series of 2,3-disubstituted pyrroles through a ring-closing metathesis protocol (**Scheme 4**).[7]

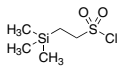
SES-NH_2 has also be used in the selective synthesis of various triazamacrocycles.[8,9] The macrocycles are constructed using a modular approach, which allows the researcher considerable control in the carbon bridge architecture of the macrocycle (**Scheme 5**).

References: 1) Weinreb, S. M.; Ralbovsky, J. L. "β-Trimethylsilylethanesulfonyl Chloride," in Encyclopedia of Reagents for Organic Synthesis; Paquette, L. A., Ed.; Wiley: Chichester, U.K., 1995, Vol. 7, p. 5255–5256. (2) Ribière, P. et al. *Chem. Rev.* **2006**, *106*, 2249. (3) (a) Rew, Y. et al. *J. Am. Chem. Soc.* **2004**, *126*, 1041. (b) Jiang, W. et al. *J. Am. Chem. Soc.* **2003**, *125*, 1877. (c) Jiang, W. et al. *J. Am. Chem. Soc.* **2002**, *124*, 5288. (4) Desai, P. et al. *Org. Lett.* **2003**, *5*, 5047. (5) Ohno, H. et al. *J. Am. Chem. Soc.* **2004**, *126*, 8744. (6) Mancheño, O. G.; Bolm, C. *Org. Lett.* **2006**, *8*, 2349. (7) Declerck, V. et al. *J. Org. Chem.* **2004**, *69*, 8372. (8) Masllorens, J. et al. *Tetrahedron* **2005**, *61*, 10105. (9) Parker, L. L. et al. *Tetrahedron* **2003**, *59*, 10165.

2-(Trimethylsilyl)ethanesulfonyl chloride

NEW

$C_5H_{13}ClO_2SSi$
FW: 200.76
[*106018-85-3*]

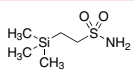


681334-1G	1 g
681334-5G	5 g

2-(Trimethylsilyl)ethanesulfonamide

NEW

$C_5H_{15}NO_2SSi$
FW: 181.33
[*125486-96-6*]



681326-1G	1 g

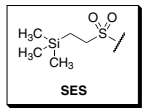
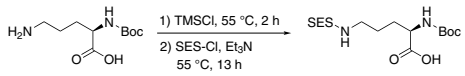
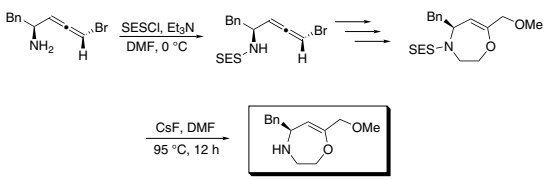


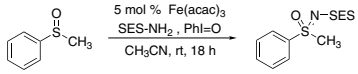
Figure 1



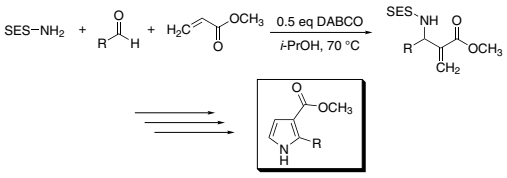
Scheme 1



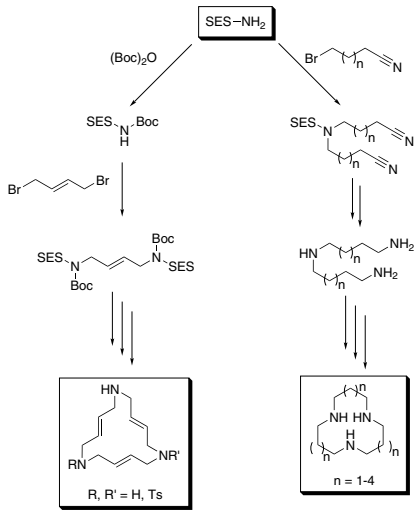
Scheme 2



Scheme 3



Scheme 4



Scheme 5

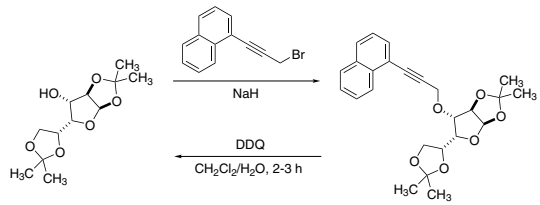
Ethynylnaphthalenes

Recent work by David Crich has been focused on the application of propargyl ethers as sterically unobtrusive donor protecting groups for β-mannosylation.[1] The propargyl ethers were easily introduced, and produced the desired effect on the stereoselectivity of the β-mannosylation reaction; however, they required an unwieldly two-step deprotection requiring the use of OsO_4. Further research elucidated the use of 3-(1-naphthyl)-2-propynyl ethers as a protecting group that retained the advantages of the propargyl ethers, yet were cleavable in a single step, orthogonal to benzyl ethers.[2]

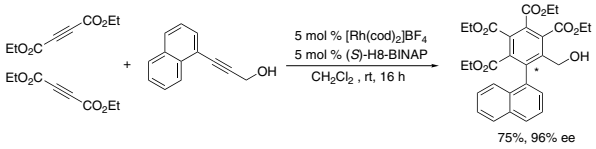
Hydroxy groups on a carbohydrate are easily alkylated with 1-(3-bromo-1-propynyl)naphthalene and sodium hydride to yield the propargyl ether. Subsequent deprotection is easily achieved by treatment with DDQ in wet dichloromethane over the course of 2-3 hours (**Scheme 1**). Furthermore, the propargyl ether proved to be exceptionally β-selective in glycosylations.

The hydroxy precursor, 3-(1-naphthyl)-2-propyn-1-ol is also useful as a building block. For example, a report from Tanaka details the use of 3-(1-naphthyl)-2-propyn-1-ol in a rhodium-catalyzed cross-cyclotrimerization with dimethyl acetylenedicarboxylate to furnish the axially chiral biaryl in good yield and excellent enatioselectivity (**Scheme 2**).[3]

References: (1) (a) Crich, D.; Jayalath, P. *Org. Lett.* **2005**, *7*, 2277. (b) Crich, D. et al. *J. Org. Chem.* **2006**, *71*, 3064. (2) Crich, D.; Wu, B. *Org. Lett.* **2006**, *8*, 4879. (3) Tanaka, K. et al. *Org. Lett.* **2005**, *7*, 3119.



Scheme 1

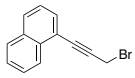


Scheme 2

1-(3-Bromo-1-propynyl)naphthalene

NEW

$C_{13}H_9Br$
FW: 245.11
[*352035-98-4*]

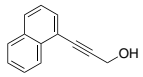


682756-1G	1 g

3-(1-Naphthyl)-2-propyn-1-ol

NEW

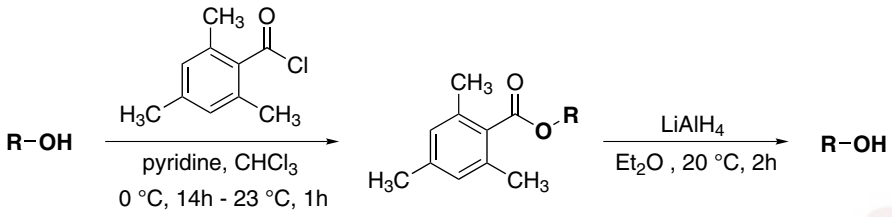
$C_{13}H_{10}O$
FW: 182.22
[*16176-22-0*]



682764-1G	1 g
682764-5G	5 g

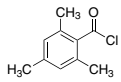
2,4,6-Trimethylbenzoyl chloride

Whereas acid chlorides might typically be thought of as building blocks, they can also be used to protect alcohols as their corresponding esters. 2,4,6-Trimethylbenzoyl chloride is a particularly useful acid chloride in this regard, as it forms the mesitoate esters. These esters are very stable to base hydrolysis, yet can be easily cleaved reductively with $LiAlH_4$.[1,2]



2,4,6-Trimethylbenzoyl chloride

$C_{10}H_{11}ClO$
FW: 182.65
[*938-18-1*]



682519-1G	1 g
682519-5G	5 g

References: (1) (a) Corey, E. J. et al. *J. Am. Chem. Soc.* **1969**, *91*, 4318. (b) Bolton, I. J. et al. *J. Chem. Soc. C* **1971**, 2944. (2) Greene, T. W.; Wuts, P. G. M. In *Protective Groups in Organic Synthesis*, 3rd ed.; John Wiley & Sons: New York, 1999; p.178–179.

Fluorous Protecting Groups[1]

Fluorous protecting groups are able to serve multiple purposes at the same time. While acting as a regular protecting group for a specific functional group they also offer an ideal opportunity to introduce a temporary fluorous tag into a molecule. This tag can be carried through a multi-step synthesis procedure, and can facilitate product workup and purification after every synthetic step.

As a general guideline, fluorous-tagged products with high fluorine content (heavy fluorous products) are recommended for natural products or medicinal chemistry synthesis in combination with either liquid-liquid or fluorous solid-phase extraction (F-SPE). Homologous fluorous-tagged products with lower fluorine content (light fluorous compounds) are useful in either fluorous chromatography or fluorous mixture synthesis.[2,3] Products with smaller fluorous tags show increased solubility in organic solvents; therefore, fluorous solvents are not necessary during the reaction, and the fluorous phase (either solid or liquid) can be used only in the separation step. At the final stage of the synthesis the desired target molecule is deprotected by the same methods as the non-fluorous analogue.

F-Boc-ON

F-Boc-ON is the fluorous equivalent of 2-(*tert*-butoxycarbonyl-oxyimino)-2-phenylacetonitrile (Boc-ON) used in protecting amino groups in peptide synthesis or other functionalities in multi-step organic synthesis. Protection of the amino group with F-Boc-ON and deprotection are achieved under traditional reaction conditions, with the advantage that products containing the F-Boc group can easily be separated from organic reagents, reactants, or products by performing a quick fluorous solid-phase extraction over Fluoro*Flash*® Silica Gel.[4]

In a recent example, F-Boc-ON was used for the preparation and isolation of DNA minor groove binding polyamides containing *N*-methylpyrrole. The authors found that the isolation of the fluorous intermediates in the synthetic scheme was advantageous when compared to conventional purification methods (**Scheme 1**).[5]

F-PMB-OH

F-PMB-OH is the fluorous equivalent of *p*-methoxybenzyl alcohol (PMB-OH) used in protecting alcohols in multi-step organic synthesis. F-PMB can be deprotected either under typical acid or oxidizing conditions. The reactivity of F-PMB and conventional PMB are so similar that a 3,4-dimethoxybenzyl (DMB) protecting group has been selectively cleaved in the presence of both F-PMB and PMB (**Scheme 2**).[6]

F-Trityl Alcohol and Chlorides

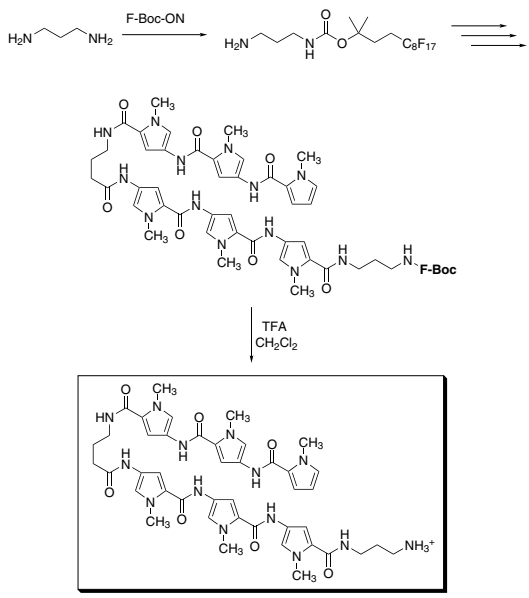
Several fluorous versions of trityl protecting groups are available from Sigma-Aldrich for the protection of alcohols, amines and carboxylates. These include F-Trt, F-MMT, and F-DMT that are analogous to the conventional trityl, monomethoxy trityl, and dimethoxy trityl groups and react analogously to their non-fluorous counterpart. Each of these groups is acid labile with the relative rate of deprotection being F-Trt < F-MMT < F-DMT. The F-DMT group has recently been used in oligonucleotide synthesis followed by purification through F-SPE.[7]

F-Silanes

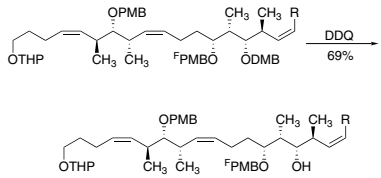
F-Silanes are the fluorous equivalent to a TIPS group. They exhibit properties similar to most silicon protecting groups and have been used in both parallel and fluorous mixture synthesis.[8,9] Tagging of an alcohol is accomplished by in situ activation of the F-silane to either the bromide or triflate followed by addition of the alcohol. Detagging of the F-silane is accomplished either by treatment with fluoride or acid.

F-Benzyl Alcohol, F-Fmoc-Cl, F-Z-OSu

These reagents are the fluorous equivalents of their parent protecting groups and are fully compatible with the corresponding protection and deprotection methods conventionally associated with that group.



Scheme 1

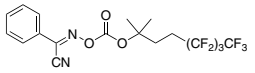


Scheme 2

F-Protecting Groups

2-[(4,4,5,5,6,6,7,7,7-Nonafluoro-1,1-dimethylheptyloxy) carbonyloxyimino]-2-phenylacetonitrile, ≥ 97.0%

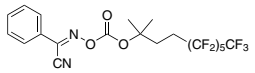
$C_{18}H_{15}F_9N_2O_3$
FW: 478.31



01382-1G-F	1 g

2-[(4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluoro-1,1-dimethyl nonyloxy)carbonyloxyimino]-2-phenylacetonitrile, ≥ 97.0%

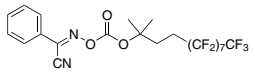
$C_{20}H_{15}F_{13}N_2O_3$
FW: 578.32



11807-1G-F	1 g
11807-5G-F	5 g

2-[(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-1,1-dimethylundecyloxy)carbonyloxyimino]-2-phenylacetonitrile, ≥ 97.0%

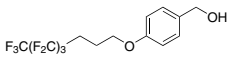
$C_{22}H_{15}F_{17}N_2O_3$
FW: 678.34
[*350716-42-6*]



55118-1G-F	1 g
55118-5G-F	5 g

4-(4,4,5,5,6,6,7,7,7-Nonafluoroheptyloxy)benzyl alcohol, ≥ 97.0%

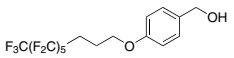
$C_{14}H_{13}F_9O_2$
FW: 384.24



01452-1G-F	1 g

4-(4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluorononyloxy) benzyl alcohol, ≥ 97.0%

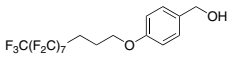
$C_{16}H_{13}F_{13}O_2$
FW: 484.25



67772-1G-F	1 g
67772-5G-F	5 g

4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecyloxy)benzyl alcohol, ≥ 97.0%

$C_{18}H_{13}F_{17}O_2$
FW: 584.27

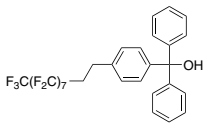


97071-1G-F	1 g
97071-5G-F	5 g

1-[4-(1*H*,1*H*,2*H*,2*H*-Perfluorodecyl)phenyl]-1,1-diphenylmethanol, 98%

NEW

$C_{29}H_{19}F_{17}O$
FW: 706.43
[*649561-66-0*]

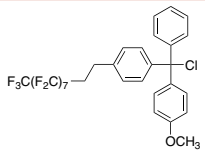


672475-1G	1 g

1-(4-Methoxyphenyl)-1-[4-(1*H*,1*H*,2*H*,2*H*-perfluorodecyl)phenyl]-1-phenylmethyl chloride

NEW

$C_{30}H_{20}ClF_{17}O$
FW: 754.91
[*865758-37-8*]

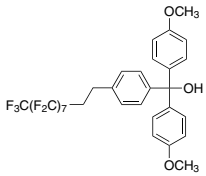


672149-1G	1 g

1,1-Di-(4-methoxyphenyl)-1-[4-(1*H*,1*H*,2*H*,2*H*-perfluorodecyl)phenyl]methanol, 97%

NEW

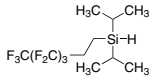
$C_{31}H_{23}F_{17}O_3$
FW: 766.49
[*865758-47-0*]



672696-1G	1 g

Diisopropyl(3,3,4,4,5,5,6,6,6-nonafluorohexyl)silane, ≥ 95%

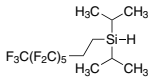
$C_{12}H_{19}F_9Si$
FW: 362.35
[*356056-13-8*]



18976-1G-F	1 g

Diisopropyl(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl) silane, ≥ 95%

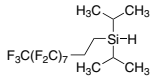
$C_{14}H_{19}F_{13}Si$
FW: 462.37
[*356056-14-9*]



00454-1G-F	1 g
00454-5G-F	5 g

Diisopropyl(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadeca fluorodecyl)silane, ≥ 95%

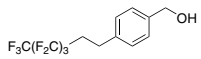
$C_{16}H_{19}F_{17}Si$
FW: 562.38
[*356056-15-0*]



04537-1G-F	1 g
04537-5G-F	5 g

4-(3,3,4,4,5,5,6,6,6-Nonafluorohexyl)benzyl alcohol, ≥ 95%

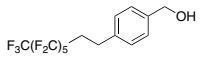
$C_{13}H_{11}F_9O$
FW: 354.21



08431-1G-F	1 g

4-(3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctyl)benzyl alcohol, ≥ 97.0%

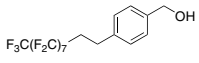
$C_{15}H_{11}F_{13}O$
FW: 454.23
[*356055-76-0*]



16638-1G-F	1 g
16638-5G-F	5 g

4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecyl) benzyl alcohol, ≥ 98.0%

$C_{17}H_{11}F_{17}O$
FW: 554.24
[*356055-77-1*]

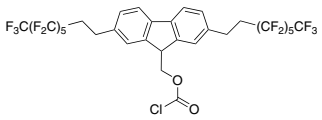


19563-1G-F	1 g
19563-5G-F	5 g

2,7-Bis(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-9-fluorenylmethoxycarbonyl chloride, 98%

NEW

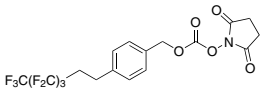
$C_{31}H_{17}ClF_{26}O_2$
FW: 950.88



672262-1G	1 g

N-[4-(3,3,4,4,5,5,6,6,6-Nonafluorohexyl)benzyl oxycarbonyloxy]succinimide, ≥ 95.0%

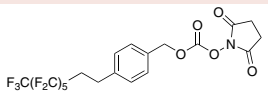
$C_{18}H_{14}F_9NO_5$
FW: 495.29



00246-1G-F	1 g

N-[4-(3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctyl)benzyl oxycarbonyloxy] succinimide, ≥ 97.0%

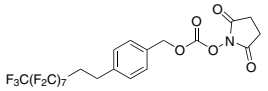
$C_{20}H_{14}F_{13}NO_5$
FW: 595.31
[*556050-48-7*]



05656-1G-F	1 g
05656-5G-F	5 g

N-[4-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadeca-fluorodecyl) benzyloxycarbonyloxy]succinimide, ≥ 97.0%

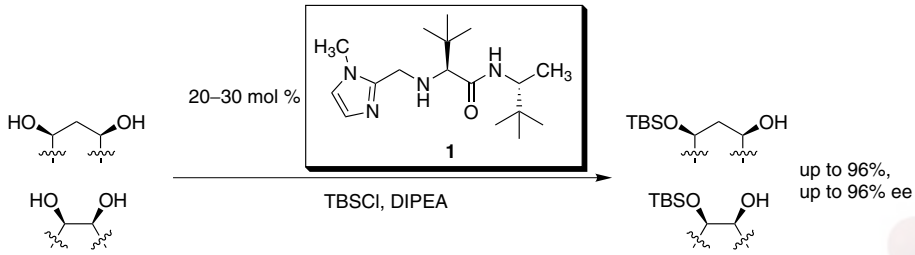
$C_{22}H_{14}F_{17}NO_5$
FW: 695.32
[*556050-49-8*]



14944-1G-F	1 g
14944-5G-F	5 g

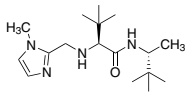
Hoveyda-Snapper Catalyst For Desymmetrization Of *meso*-Diols

Professors Marc Snapper and Amir Hoveyda at Boston College have recently reported[1] the development of an amino-acid-based small molecule **1** capable of promoting asymmetric monosilylation of *meso*-1,2-diols. The catalyst is compatible with a variety of silyl chlorides and generally provides enantioselectivities above 88%, and the reactions do not require rigorous exclusion of air or moisture. Furthermore, the catalyst can be easily recovered in near-quantitative yield after use and subsequently reused with identical efficiency. This catalyst greatly increases the efficiency with which optically-enriched molecules can be prepared.



(*S*)-*N*-[(*R*)-3,3-Dimethyl-2-butyl]-3,3-dimethyl-2-[(1-methyl-1*H*-imidazol-2-yl)methylamino]butanamide

$C_{17}H_{32}N_4O$
FW: 308.46



680826-1G	1 g

Reference: (1) Zhao, Y. et al. *Nature* **2006**, *443*, 67.

Fluorous Separation Media

The separation of fluorous compounds by F-SPE (Fluorous Solid Phase Extraction) is a reliable and generic procedure that more greatly resembles filtration than chromatography. The quick separation depends primarily on the presence or absence of a light fluorous tag, not polarity or other molecular features that control traditional chromatography.[10] Fluoro*Flash*® silica selectively retains fluorous molecules while non-fluorous compounds are not retained. The fluorous compounds can then be recovered easily through a simple solvent change. Fluoro*Flash*® SPE cartridges are pre-packed in a variety of formats with a proprietary fluorous silica gel.

In **Figure 1**, a mixture of a non-fluorous dye (blue) and a fluorous dye (orange) are loaded on a fluorous adsorbent (left-hand test tube). The non-fluorous dye can be washed down with a fluorophobic solvent mixture like aqueous methanol (middle test tube). The fluorous dye remains on the adsorbent until the elution with a fluorophilic wash (e.g. with pure methanol, right-hand test tube). The colored dyes of this example were used to visualize the easy separation of a fluorous-tagged product from non-fluorous byproducts with F-SPE (Fluorous Solid Phase Extraction).

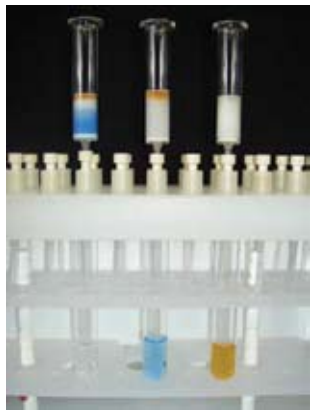


Figure 1

References: (1) The fluorous products featured here are manufactured by Fluorous Technologies, Inc. U.S. patents 6,156,896; 5,859,247; 5,777,121 and 6,673,539 may protect use of these compounds. (2) Zhang, Q. et al. *J. Am. Chem. Soc.* **2004**, *126*, 36. (3) Curran, D. P.; Oderaotoshi, Y. *Tetrahedron* **2001**, *57*, 5243. (4) Curran, D. P. *Synlett* **2001**, 1488. (5) Mamidyala, S. K.; Firestine, S. M. *Tet. Lett.* **2006**, *47*, 7431. (6) Curran, D. P.; Furukawa, T. *Org. Lett.* **2002**, *4*, 2233. (7) Pearson, W. H. et al. *J. Org. Chem.* **2005**, *70*, 7114. (8) Palmacci, E. R. et al. *Angew. Chem. Int. Ed.* **2001**, *40*, 4433. (9) Zhang, W. et al. *J. Am. Chem. Soc.* **2002**, *124*, 10443. (10) Zhang, W.; Curran, D.P. *Tetrahedron* **2006**, *62*, 11837.

Fluorous Separation Media

Fluoro*Flash*® SPE Cartridges, 2 grams, 8 cc tube, particle size 40µm
14196-1EA-F

Fluoro*Flash*® SPE Cartridges, 5 grams, 10 cc tube, particle size 40µm
00866-1EA-F

Fluoro*Flash*® SPE Cartridges, 10 grams, 60 cc tube, particle size 40µm
08967-1EA-F

Fluoro*Flash*® SPE Cartridges, 20 grams, 60 cc tube, particle size 40µm
08966-1EA-F

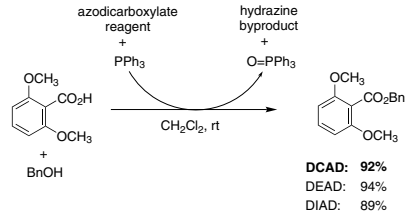
Fluoro*Flash*® SPE Cartridges, 20 grams, 60 cc tube, particle size 40µm
06961-1EA-F

Fluoro*Flash*® TLC Plates, with F254 indicator
16888-1EA-F

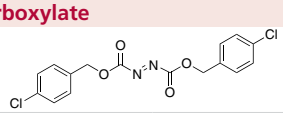
Fluoro*Flash*® Silica Gel 40 µm, particle size ~40µm
08965-1EA-F

Lipshutz DCAD Coupling Reagent

The Mitsunobu reaction is one of the most extensively used coupling reactions in organic synthesis and typically azodicarboxylate reagents such as DEAD or DIAD are employed. However, drawbacks such as low room temperature stability and difficulty in removing hydrazine byproducts detract from the practicability of these reagents. Recent work by Professor Bruce Lipshutz and co-workers details the development of a new azodicarboxylate for the Mitsunobu reaction.[1] DCAD is a solid reagent and can easily be stored and handled at room temperature, unlike the more common DEAD and DIAD reagents. DCAD exhibits comparable reactivity to DEAD in common Mitsunobu couplings; however, unlike DEAD, the reduced hydrazine byproduct of DCAD is easily removed by simple precipitation directly from the reaction mixture, and is easily recycled in high yield to regenerate DCAD.



Di-(4-chlorobenzyl) azodicarboxylate
DCAD
$C_{16}H_{12}Cl_2N_2O_4$
FW: 367.18



680850-1G	1 g
680850-10G	10 g

References: (1) Lipshutz, B. H. et al. *Org. Lett.* **2006**, *8*, 5069.

Common Reagents for Protection

TMS

Chlorotrimethylsilane, redistillation, ≥ 99%

TMSCl
C_3H_9ClSi
FW: 108.64
[*75-77-4*]

386529-100ML	100 mL
386529-1L	1 L

Bromotrimethylsilane, 97%

TMSBr
C_3H_9BrSi
FW: 153.09
[*2857-97-8*]

194409-5G	5 g
194409-25G	25 g
194409-100G	100 g

Iodotrimethylsilane, 97%

TMSI
C_3H_9ISi
FW: 200.09
[*16029-98-4*]

195529-5G	5 g
195529-25G	25 g
195529-100G	100 g

Trimethylsilyl trifluoromethanesulfonate, 99%

TMS(OTf)
$C_4H_9F_3O_3SSi$
FW: 222.26
[*27607-77-8*]

225649-10G	10 g
225649-50G	50 g

N,O-Bis(trimethylsilyl)acetamide, synthesis grade

BSA
$C_8H_{21}NOSi_2$
FW: 203.43
[*10416-59-8*]

128910-10ML	10 mL
128910-25ML	25 mL
128910-100ML	100 mL
128910-1L	1 L

N,O-Bis(trimethylsilyl)trifluoroacetamide, ≥ 99%

BSTFA
$C_8H_{18}F_3NOSi_2$
FW: 257.4
[*25561-30-2*]

155195-5G	5 g
155195-25G	25 g
155195-100G	100 g

N-Methyl-*N*-(trimethylsilyl)trifluoroacetamide, derivatization grade

MSTFA
$C_6H_{12}F_3NOSi$
FW: 199.25
[*24589-78-4*]

394866-5ML	5 mL
394866-10X1ML	10 mL
394866-25ML	25 mL

TBDMS

tert-Butyldimethylsilyl chloride, reagent grade, 97%

TBDMSCl
$C_6H_{15}ClSi$
FW: 150.72
[*18162-48-6*]

190500-5G	5 g
190500-25G	25 g
190500-100G	100 g
190500-1KG	1 kg
190500-10KG	10 kg

tert-Butyldimethylsilyl trifluoromethanesulfonate, reagent grade, 98%

TBDMS(OTf)
$C_7H_{15}F_3O_3SSi$
FW: 264.34
[*69739-34-0*]

226149-1G	1 g
226149-5G	5 g
226149-25G	25 g

N-tert-Butyldimethylsilyl-*N*-methyltrifluoroacetamide, > 97%

MTBSTFA
$C_9H_{18}F_3NOSi$
FW: 241.33
[*77377-52-7*]

394882-5ML	5 mL
394882-10X1ML	10 mL
394882-25ML	25 mL
394882-100ML	100 mL

TES

Chlorotriethylsilane, 99%

TESCl
$C_6H_{15}ClSi$
FW: 150.72
[*994-30-9*]

235067-5G	5 g
235067-25G	25 g

TIPS

Triisopropylsilyl chloride, 97%

TIPSCl
$C_9H_{21}ClSi$
FW: 192.8
[*13154-24-0*]

241725-10G	10 g
241725-50G	50 g

Triisopropylsilyl trifluoromethanesulfonate, 97%

TIPS(OTf)
$C_{10}H_{21}F_3O_3SSi$
FW: 306.42
[*80522-42-5*]

248460-10G	10 g
248460-50G	50 g

TBDPS

tert-Butyl(chloro)diphenylsilane, 98%

TBDPSCl
$C_{16}H_{19}ClSi$
FW: 274.86
[*58479-61-1*]

195537-2G	2 g
195537-10G	10 g
195537-50G	50 g

DTBS

Di-*tert*-butylsilyl bis(trifluoromethanesulfonate), 97%

$DTBS(OTf)_2$
$C_{10}H_{18}F_6O_6S_2Si$
FW: 440.45
[*85272-31-7*]

262021-5G	5 g
262021-25G	25 g

Boc

Di-*tert*-butyl dicarbonate, reagent grade, 97%

Boc anhydride
$C_{10}H_{18}O_5$
FW: 218.25
[*24424-99-5*]

199133-25G	25 g
199133-100G	100 g

Di-*tert*-butyl dicarbonate, *ReagentPlus*®, 99%

Boc anhydride
$C_{10}H_{18}O_5$
FW: 218.25
[*24424-99-5*]

205249-10G	10 g
205249-50G	50 g
205249-100G	100 g
205249-1KG	1 kg
205249-15KG	15 kg

2-(Boc-oxyimino)-2-phenylacetonitrile, 99%

Boc-ON
$C_{13}H_{14}N_2O_3$
FW: 246.26
[*58632-95-4*]

193372-5G	5 g
193372-25G	25 g
193372-100G	100 g

Fmoc

Fmoc chloride, 97%

Fmoc-Cl
$C_{15}H_{11}ClO_2$
FW: 258.7
[*28920-43-6*]

160512-1G	1 g
160512-5G	5 g
160512-25G	25 g

Z/Cbz

Benzyl chloroformate, technical grade, 95%

Z-Cl
$C_8H_7ClO_2$
FW: 170.59
[*501-53-1*]

119938-5G	5 g
119938-100G	100 g

Dibenzyl dicarbonate, 97%

Z_2O
$C_{16}H_{14}O_5$
FW: 286.28
[*31139-36-3*]

311219-1G	1 g
311219-5G	5 g
311219-25G	25 g

Bn

Benzyl chloride, *ReagentPlus*®, 99%

BnCl
C_7H_7Cl
FW: 126.58
[*100-44-7*]

185558-50G	50 g
185558-250G	250 g
185558-1KG	1 kg
185558-2KG	2 kg
185558-4KG	4 kg

Bz

Benzoyl chloride, *ReagentPlus*®, 99%

BzCl
C_7H_5ClO
FW: 140.57
[*98-88-4*]

320153-1L	1 L

MOM

Chloromethyl methyl ether, technical grade

MOM-Cl
C_2H_5ClO
FW: 80.51
[*107-30-2*]

100331-25G	25 g
100331-100G	100 g

MEM

2-Methoxyethoxymethyl chloride, technical grade

MEM-Cl
$C_4H_9ClO_2$
FW: 124.57
[*3970-21-6*]

357480-5G	5 g
357480-25G	25 g

SEM

2-(Trimethylsilyl)ethoxymethyl chloride, technical grade

SEM-Cl
$C_6H_{15}ClOSi$
FW: 166.72
[*76513-69-4*]

238902-1G	1 g
238902-5G	5 g
238902-25G	25 g

TMSE

2-(Trimethylsilyl)ethanol, 99%

TMSE-OH
$C_5H_{14}OSi$
FW: 118.25
[*2916-68-9*]

226890-1G	1 g
226890-10G	10 g
226890-50G	50 g

Ms

Methanesulfonyl chloride, ≥ 99.7%

MsCl
CH_3ClO_2S
FW: 114.55
[*124-63-0*]

471259-5ML	5 mL
471259-100ML	100 mL
471259-500ML	500 mL
471259-1L	1 L

Ts

p-Toluenesulfonyl chloride, *ReagentPlus*®, ≥ 99%

TsCl
$C_7H_7ClO_2S$
FW: 190.65
[*98-59-9*]

240877-5G	5 g
240877-100G	100 g

Trt

Trityl chloride, 98%

Trt-Cl
$C_{19}H_{15}Cl$
FW: 278.78
[*76-83-5*]

T83801-25G	25 g
T83801-100G	100 g
T83801-500G	500 g

PMB

4-Methoxybenzyl bromide

PMB-Br
C_8H_9BrO
FW: 201.06
[*2746-25-0*]

561282-5G	5 g

THP

3,4-Dihydro-2*H*-pyran, 97%

C_5H_8O
FW: 84.12
[*110-87-2*]

D106208-5ML	5 mL
D106208-100ML	100 mL
D106208-500ML	500 mL

EtG

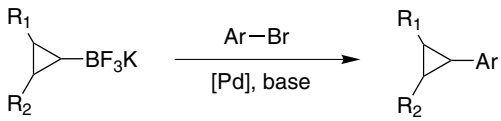
Ethylene glycol, *ReagentPlus*®, ≥ 99%

$C_2H_6O_2$
FW: 62.07
[*107-21-1*]

102466-500ML	500 mL
102466-1L	1 L
102466-6X500ML	3000 mL
102466-4L	4 L
102466-20L	20 L

Potassium Cyclopropyltrifluoroborate

Cyclopropyl groups are found in a variety of natural products and are increasingly incorporated into pharmaceuticals such as the broad-spectrum antibiotic ciprofloxacin. Both the Charette[1] and Deng[2] groups have reported success in the cross-coupling of potassium cyclopropyltrifluoroborates with aryl bromides in the presence of common palladium catalysts. The trifluoroborate salts exhibit enhanced stability and more certain stoichiometry relative to their boronic acid counterparts. However, like boronic acids, post-reaction byproducts are easily removed. We are pleased to add this useful reagent to our ever-growing portfolio of organoboron compounds.



Potassium cyclopropyltrifluoroborate

$C_3H_5BF_3K$
FW: 147.98

662984-1G	1 g
662984-5G	5 g

(1) Charette, A. B. et al. *Synlett* **2005**, *11*, 1779. (2) Fang, G.-H. et al. *Org. Lett.* **2004**, *6*, 357.

Common Reagents for Deprotection

Cesium fluoride, 99%

CsF
FW: 151.9
[*13400-13-0*]

198323-25G	25 g
198323-100G	100 g

Cesium fluoride, 99.9%

CsF
FW: 151.9
[*13400-13-0*]

289345-5G	5 g
289345-25G	25 g
289345-100G	100 g

Hydrogen fluoride pyridine, hydrogen fluoride ~70%, pyridine ~30%

HF
FW: 20.01
[*62778-11-4*]

184225-25G	25 g
184225-100G	100 g

Tetrabutylammonium fluoride hydrate, 98%

$C_{16}H_{36}FN \cdot xH_2O$
FW: 261.46 (anhydrous basis)
[*22206-57-1*]

241512-10G	10 g
241512-100G	100 g

Tetrabutylammonium fluoride solution, 1.0M tetrahydrofuran

$C_{16}H_{36}FN$
FW: 261.46
[*429-41-4*]

216143-5ML	5 mL
216143-100ML	100 mL
216143-500ML	500 mL
216143-2L	2 L

Tetramethylammonium fluoride, 97%

$C_4H_{12}FN$
FW: 93.14
[*373-68-2*]

459135-1G	1 g
459135-5G	5 g

2,3-Dichloro-5,6-dicyano-*p*-benzoquinone, 98%

$C_8Cl_2N_2O_2$
FW: 227
[*84-58-2*]

D60400-5G	5 g
D60400-10G	10 g
D60400-100G	100 g

Ammonium cerium(IV) nitrate, ACS reagent, ≥ 98.5%

$H_8CeN_8O_{18}$
FW: 548.22
[*16774-21-3*]

215473-50G	50 g
215473-250G	250 g
215473-500G	500 g

Ammonium cerium(IV) nitrate, 99.99%

$H_8CeN_8O_{18}$
FW: 548.22
[*16774-21-3*]

431338-50G	50 g
431338-250G	250 g

Boron tribromide solution, 1.0M dichloromethane

BBr_3
FW: 250.52
[*10294-33-4*]

211222-100ML	100 mL
211222-800ML	800 mL
211222-2L	2 L

Boron tribromide, *ReagentPlus*®, ≥ 99%

BBr_3
FW: 250.52
[*10294-33-4*]

419508-100G	100 g
419508-500G	500 g

p-Toluenesulfonic acid monohydrate, *ReagentPlus*®, 98.5%

$C_7H_8O_3S \cdot H_2O$
FW: 190.22
[*6192-52-5*]

T35920-5G	5 g
T35920-100G	100 g
T35920-500G	500 g

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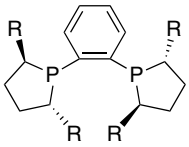
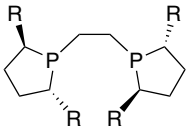
Chiral Phospholane Ligands

Chiral phospholane ligands have been used extensively in transition metal-catalyzed asymmetric hydrogenations and other novel asymmetric reactions including [4+1] cycloadditions, imine alkylations, and allylborations.

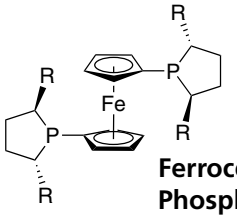
Available in either enantiomeric form with Me, Et, and *i*-Pr substituents. For a detailed product listing, visit sigma-aldrich.com/phospholane.



DuPhos



BPE



Ferrocenyl Phospholanes

Chiral phospholane ligands are sold in collaboration with Kanata Chemical Technologies Inc. for research purposes only. These compounds were made and sold under license from E. I. Du Pont de Nemours and Company, which license does not include the right to use the compounds in producing products for sale in the pharmaceutical field.

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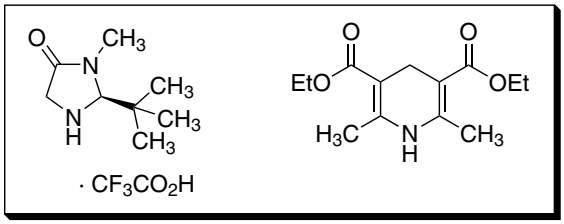

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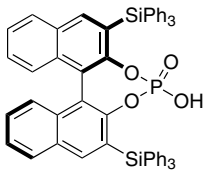
MacMillan TiPSY Catalysts have been used in the first direct enantioselective organocatalytic reductive amination reaction.

MacMillan TiPSY Catalysts



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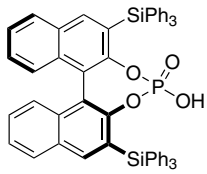

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