

ChemFiles

Supplement II, 2008

 **ALDRICH**[®]
Chemistry

FluoroFlash[®] Products for Synthesis & Separation



Sigma-Aldrich[®] and Fluorous Technologies, Inc. are partnering to bring you easier, faster, and more robust purifications.

FluoroFlash Tags for
Solution Phase Synthesis

FluoroFlash Scavengers
and Reagents

FluoroFlash SPE
Cartridges for
Fluorous Separations

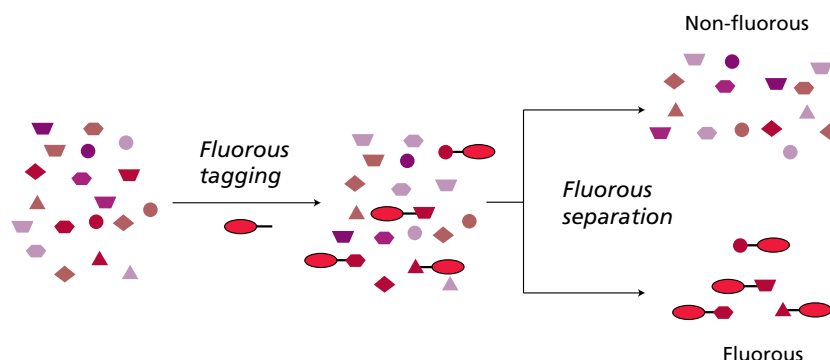
SIGMA-ALDRICH[®]

Simplify Purification with FluoroFlash® Products!

In partnership with Fluorous Technologies, Inc., Sigma-Aldrich® is pleased to offer a wide range of fluororous products that allow for faster, more robust purifications. By combining fluororous tags with fluororous solid phase extraction (F-SPE), researchers can quickly and efficiently purify complex chemical mixtures. In this brochure you will find a representative listing of applications and FluoroFlash® products available through Sigma-Aldrich at research scale to support this important new separation technology.

General Concept:

Fluorous chemistry is a novel tagging and separation technology based on the use of perfluoroalkyl (fluorous) domains. Fluorous-tagged molecules can be readily separated from non-fluorous molecules exploiting fluorophilicity.



Features and Benefits of Fluorous Chemistry:

- Complete solution phase chemistry – highly adaptable
- Orthogonal mechanism of separation – very selective
- Based on fluorinated materials – chemically inert
- Applicable to numerous molecular classes – broad utility
- Compatible with standard lab equipment – easily integrated

Key Applications:

- Medicinal Chemistry
- Parallel Synthesis
- Combinatorial Chemistry
- Catalysis
- Peptide & Carbohydrate Synthesis
- Microwave-assisted Organic Synthesis

Useful References:

Besides the information in this brochure, you can learn much more about fluororous techniques at the Sigma-Aldrich website portal: sigma-aldrich.com/fluorous. There you will find application notes, ChemFiles, a Cheminar™ on fluororous chemistry, *Aldrichimica Acta* articles and many other useful references.

Technical Support:

Fluorous Technologies, Inc. may be contacted by phone (412-826-3050) or email (contact-fti@fluorous.com) to answer any questions you may have about fluororous products and applications.



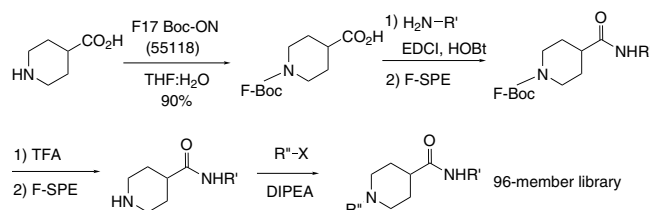
FluoroFlash® Tags for Solution Phase Synthesis

FluoroFlash® tags are perfluoroalkyl modified versions of traditional protecting groups familiar to synthetic chemists. These tags have reactivity profiles very similar to the traditional protecting group, but have the added benefit of providing a phase tag for purification. FluoroFlash tags are a solution phase alternative to solid phase synthesis strategies. The fluorinated-tagged materials are easily separated from non-tagged components by F-SPE.

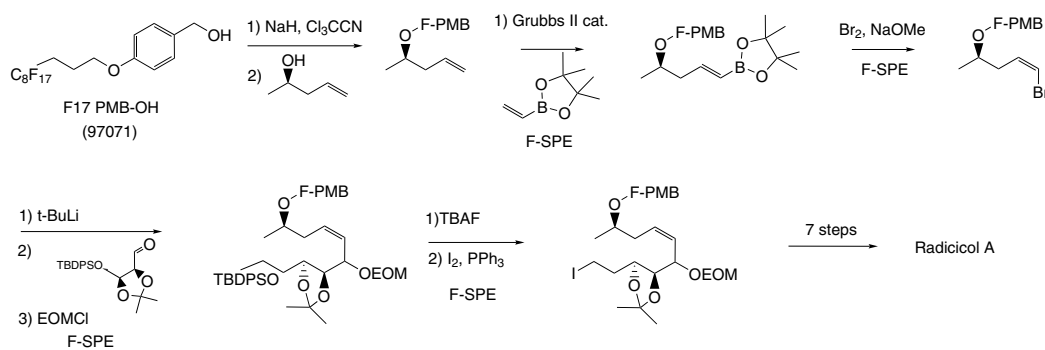
Benefits of FluoroFlash Tags:

- Solution phase kinetics
- Broad reaction compatibility
- Reaction monitoring by TLC, GC, LC, NMR, etc.
- Readily scalable

Fluorous BOC protection used in combinatorial synthesis



Application example of a fluorinated PMB protecting group used in the multistep synthesis of natural product Radicol A



Dakas, P.-Y. et al, *Angew. Chem. Intl. Ed.* **2007**, *46*, 6899

Sigma-Aldrich® offers a full line of FluoroFlash tags. Representative examples are shown in the table below. FluoroFlash tags are also available in other perfluorocarbon chain lengths. For a complete listing of available products, please visit sigma-aldrich.com/fluorous.

Product No.	Name	Structure	Product No.	Name	Structure
55118	F17 Boc-ON		19563	F17 Benzyl-OH	
04537	F17 Silane		672475	F17 Trityl-OH	
14944	F17 Cbz-OSu		40829	F17 FluorMar™	
97071	F17 PMB-OH		672262	F26 Fmoc-Cl	

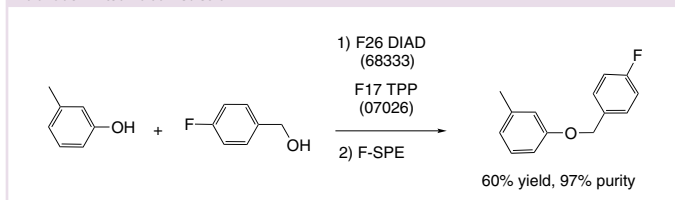
FluoroFlash® Scavengers and Reagents

FluoroFlash® scavengers and reagents are analogous to solid phase supported scavengers and reagents that are commonly used in solution phase synthesis strategies. FluoroFlash scavengers, reagents, and their byproducts can be readily removed by F-SPE.

Benefits of FluoroFlash Scavengers and Reagents:

- Solution phase kinetics
- Precise stoichiometric control
- Less molar equivalents required per reaction

Fluorous Mitsunobu reaction



Sigma-Aldrich offers a full line of FluoroFlash scavengers and reagents. Representative examples are shown in the table below. For a complete listing of available products, please visit sigma-aldrich.com/fluorous.

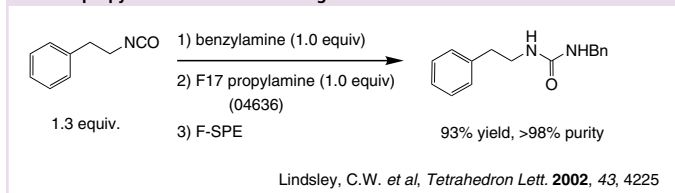
Product No.	Name	Structure
04636	F17 Propylamine	
07856	F17 Benzylamine	
08686	F17 Thiol	
07172	F17 Isatoic Anhydride	
18486	F17 Ethyl Isocyanate	
16429	F17 DAIB	

Product No.	Name	Structure
06694	F27 Tin Hydride	
07026	F17 Triphenylphosphine	
68333	F26 DIAD	
672378	F26 CDMT	

Other Fluorous Technologies Products available through Sigma-Aldrich®

- Fluorous reagents for Proteomics and Metabolomics Applications
- Additional FluoroFlash tags for Protecting Group chemistry
- FluoroFlash products for Fluorous Peptide & Carbohydrate Synthesis
- Expanded FluoroFlash Tin, Phosphine, Catalyst, and Ligand offerings
- FluoroFlash Tags for Fluorous Mixture Synthesis
- FluoroFlash Building Blocks to Design Your Custom Fluorous Tags

Fluorous propylamine used as a scavenger



For more details, please visit sigma-aldrich.com/fluorous

FluoroFlash® SPE Cartridges for Fluorous Separations

The separation of fluorous compounds from non-fluorous compounds using FluoroFlash® silica gel is the central theme of fluorous techniques. Fluorous solid phase extraction (F-SPE) is used for quick separations of reaction mixtures involving FluoroFlash tags, scavengers, and reagents. The separation of fluorous compounds by F-SPE is a reliable and generic procedure that resembles filtration more than chromatography. The facile separation depends primarily on the presence or absence of a light fluorous tag, not polarity or other molecular features that control traditional chromatography.

A simple two-step separation involves the loading of a reaction mixture containing the fluorous-tagged molecule onto an F-SPE cartridge, washing the non-fluorous compounds away using a fluorophobic wash (typically 80:20 MeOH:water), then washing off the fluorous compounds with a fluorophilic wash, such as MeOH or THF. No fluorous solvents are necessary. The cartridge can be regenerated by washing with acetone or THF. Loading capacity for F-SPE cartridges is between 5-15% by weight of fluorous silica gel.

Fluorous Solid Phase Extraction Dye Demonstration



A visual representation of the FluoroFlash solid phase extraction technique illustrates the easy separation of a fluorous-tagged product from the non-fluorous compounds through the use of colored dyes.

- A mixture of a non-fluorous dye and a fluorous dye is loaded onto the F-SPE cartridge
- Non-fluorous dye is washed off with a fluorophobic solvent wash, 80:20 MeOH:water. (middle test tube)
- Fluorous dye is washed off with a fluorophilic solvent wash, MeOH. (right test tube)

A detailed F-SPE Application Note can be found at the Sigma-Aldrich® website portal: sigma-aldrich.com/fluorous.

Benefits of FluoroFlash SPE separations:

- High selectivity
- Simple binary separation
- Large loading capacity
- No fluorous solvents needed
- Cartridges recyclable up to 10 times
- Automation compatible

Sigma-Aldrich offers FluoroFlash silica gel in pre-packed cartridges, in bulk, or as TLC plates.

Product No.	Product	
14196	FluoroFlash SPE cartridges, 2 g, 8 cc tube	Pack of 20
00866	FluoroFlash SPE cartridges, 5 g, 10 cc tube	Pack of 10
08967	FluoroFlash SPE cartridges, 10 g, 60 cc tube	Pack of 5
08966	FluoroFlash SPE cartridges, 20 g, 60 cc tube	Pack of 2
06961	FluoroFlash SPE cartridges, 20 g, 60 cc tube	Pack of 5
08965	FluoroFlash Silica Gel, 40 micron particle size	100 g
16888	FluoroFlash TLC Plates with F254 indicator	Pack of 10

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