


Table 1: F-SPE CARTRIDGES LOADING CAPACITY

Size	Size	Size
2 g, 8 mL tube	801-0027S	100-300 mg
5 g, 10 mL tube	801-0058S	400-600 mg
10 g, 60 mL tube	801-0109S	0.5-1.5 g
20 g, 60 mL tube	801-0209A	1.5-2.5 g

WHAT'S IN FluoroFlash® CARTRIDGES? Fluorous solid-phase extraction (F-SPE) cartridges are packed with silica gel containing a perfluorooctylethylsilyl ($\text{Si}(\text{CH}_2)_2\text{C}_8\text{F}_{17}$) bonded phase. Fluorous silica separates compounds based primarily on fluorine content. It selectively retains fluorous molecules while non-fluorous organic compounds, regardless of polarity, are not retained.

WHAT'S THE UTILITY OF FLUOROUS SPE (F-SPE)? F-SPE is used for quick separation of reaction mixtures involving fluorous reagents,^{1,2} protecting groups,³ tags⁴ and scavengers.⁵ In a simple two-step elution, fluorous compounds and non-fluorous compounds are separated into two fractions.

GENERAL PROCEDURES FOR F-SPE: Following completion of a reaction involving a fluorous-tagged molecule, the reaction mixture is loaded onto an F-SPE cartridge. Using a fluorophobic wash such as 80:20 MeOH:H₂O, the non-fluorous organic compounds are washed off the cartridge. The fluorous compounds are then washed off the cartridge using a fluorophilic second wash such as MeOH, acetone or THF. The loading capacity for fluorous SPE is typically between 5-10% by weight of fluorous silica gel. The cartridge can be regenerated by washing thoroughly with acetone or THF and reused multiple times (See FAQ #13).

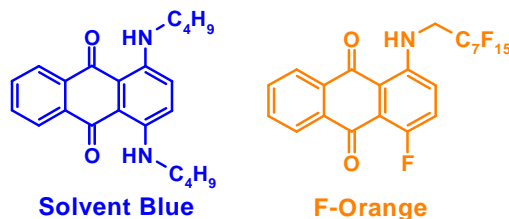
DEMONSTRATION WITH DYES: The following dye separation demonstrates how F-SPE works. The non-fluorous compound is Solvent Blue® Dye while the fluorous compound is F-Orange Dye.

Figure 1 shows fluorous cartridges containing the dye mixture in three different stages of elution. The left-hand test-tube illustrates how the F-SPE cartridge appears after loading a mixture of the two dyes and elution with a small amount of 80:20 MeOH:H₂O. The center tube shows how the non-fluorous components (blue fraction) are washed from the cartridge by using more 80:20 MeOH:H₂O. The adsorbed fluorous dye is not eluted even with extensive flushing with 80:20 MeOH:H₂O and remains on the cartridge. Finally, the orange fluorous dye is easily eluted out with 100% MeOH or THF, as shown by the third tube.

Multiple fraction collection and analysis are not required—there is one organic wash and one fluorous wash. Since the separation process is highly reproducible and functional group independent, it can be easily automated or used as plate-to-plate format.



Figure 1. F-SPE with Blue (Organic) and Orange (Fluorous) Dyes



Left tube: beginning of fluorophobic wash (80:20 MeOH:H₂O)

Center tube: end of fluorophobic wash

Right tube: end of fluorophilic wash (100% MeOH)

TYPICAL F-SPE PROCEDURE (For separation of a reaction mixture (100-300 mg) in 0.5 mL of DMF with a 2g SPE cartridge):

- Step 1 - Wash the new cartridge:** Wash a new cartridge with 1 mL of DMF under a vacuum or positive pressure depending on your SPE manifold. This step can be omitted with recycled cartridges.
- Step 2 - Preconditioning:** Pass through 6 mL of 80:20 MeOH:H₂O to condition the cartridge. Discard the preconditioning eluent.
- Step 3 - Sample loading:** Dissolve sample (100-300 mg) in 0.5 mL of DMF and load onto the cartridge using vacuum or positive pressure to ensure that the sample is completely adsorbed onto the cartridge (see Table 2 for alternative loading solvents).
- Step 4 - Fluorophobic elution:** Wash with 6-8 mL of 80:20 MeOH:H₂O to obtain the fraction containing the organic compounds.
- Step 5 - Fluorophilic elution:** Wash with 8 mL of MeOH to obtain the fraction containing the fluorous compounds.
- Step 6 - Regenerate the cartridge:** To regenerate the SPE cartridge, wash with 6-10mL of THF or acetone before reuse. Then follow Steps 2-5.

 Table 2: Suggested Maximum Loading Solvent Volumes for C₈F₁₇ Tagged Substrates

Solvent	Maximum Loading Volume		
	2 g cartridge	5 g cartridge	10 g cartridge
THF	0.2 mL	0.5 mL	1.0 mL
CH ₂ Cl ₂	0.2 mL	0.5 mL	1.0 mL
MeOH	0.2 mL	0.5 mL	1.0 mL
DMF	0.8mL	2.0 mL	4.0 mL
DMF/H ₂ O (9:1)	2.0mL	5.0mL	10mL
DMSO	2.0mL	5.0mL	10mL

Please contact FTI for information about the use of other solvents with F-SPE

