



Positive Pressure Plate-to-Plate F-SPE

General Information:

- The manifold can be purchased through Chrom Tech, Inc. www.chromtech.com.
- 24-, 48- or 96-well plates are packed with 3, 1, or 0.5 g fluororous silica gel (40-63 μm) in each well, respectively.
- 24-, 48- or 96-well plates are used as receiving vessels.
- The mass loading is up to 10% of silica gel. However, **5% or less is recommended**.
- Set the pressure to 15 psi. For wash and conditioning, use max flow. For elution, use flow meter to control the flow to 1 cm/min.

DESCRIPTION:

Positive pressure plate-to-plate F-SPE is developed for parallel purification of fluororous reaction mixtures. It has a standard plate format and is an element of the sets of F-SPE technologies¹.

NOTE: Before conducting F-SPE, please read the FTI Application Notes and Publications for general information on F-SPE ([F-SPE Cartridges and Frequently Asked Questions on F-SPE](#)).

96-Well F-SPE procedure:

1. Add 0.3 mL of DMF to your sample. Sonicate or shake until a solution or suspension formed.
2. Wash the silica gel with THF (1 mL), DMF (1 mL) and MeOH (1 mL) sequentially.
3. Condition the F-SPE plate with **elution solvent** (8:2 MeOH/H₂O or 9:1 DMF/H₂O) 2x1mL. Flush the solvent out.
4. Load the sample solution or suspension onto the F-SPE plate. Apply pressure for 10 seconds. Repeat if any solvent left above frits. Collect the eluent.
5. Rinse your sample vials with 1.25 mL of **elution solvent**, load onto F-SPE plate and collect the eluent. This is the 1st fraction.
6. Change the receiving plate. Wash the plate with 1.25 mL of **elution solvent**. Discard the eluent.
7. Change the receiving plate. Wash the plate with 1.25 mL of THF or acetone. This is the 2nd fraction.
8. Repeat step 6 and collect the 3rd fraction.
9. Wash the plate with THF/MeOH/TFA (1:1:0.01) 5x1 mL and dry. You can reuse the plate immediately.
10. Depending on your desired products, analyze either organic (1st) or fluororous fractions (2nd-3rd). Concentrate desired fractions and combine the products.

Notes:

1. For 24- and 48-well F-SPE, use 0.8 and 0.5 mL of DMF as loading solvent and triple or double the amount of washing solvent respectively.
2. If your desired product is the non-fluororous component (using fluororous reagents or scavengers), step 6-8 can be skipped.
3. If your desired product is the fluororous component (fluororous tags or protecting groups), THF or acetone is recommended for elution, and the flow should be controlled to <1 drop/sec. Otherwise, cross-contamination may occur due to foaming.
4. Use fresh THF. Otherwise, the products could be contaminated by peroxides.

References:

1. Zhang, W.; Curran, D.P. *Tetrahedron* **2006**, 62, 11837-11865.