Vacuum-Driven Plate-to-Plate F-SPE

General Information:

- Manifolds for vacuum-driven SPE can be purchased from many vendors.
- 24- and 48-well plates are packed with 3 g and 1 g fluorous silica gel (40-63 μm) in each well, respectively. 3 g and 1 g cartridges can also be used for some manifolds.
- Depending on manifolds, 24- and 48-well plates or test tubes are used as receiving vessels.
- The mass loading is up to 10% of silica gel, however, **5% or less is recommended**.
- For wash and conditioning, use max flow. For elution, control the vacuum to 5-10 inch Hg so that the flow is <1 in/min.

DESCRIPTION:

Vacuum-driven plate-to-plate F-SPE is developed for parallel purification of fluorous reaction mixtures. It has a standard plate format and is an element of the sets of F-SPE technologies[1-2]

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)

PROCEDURE:

24-well F-SPE procedure:

1. Add 0.8 mL of DMF to each sample. Sonicate or shake until a solution or suspension formed.
2. Wash the silica gel with THF (5 mL), DMF (5 mL) and MeOH (5 mL) sequentially.
3. Condition the F-SPE plate with your **elution solvent** (8:2 MeOH/H_2O or 9:1 DMF/H_2O) 2x5 mL. Flush the solvent out.
4. Load the sample solutions or suspensions onto the F-SPE plate. Apply vacuum until no solvent left above frits. Collect the eluents.
5. Rinse the sample vials with 4.5 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 5 mL of **elution solvent**. This is the 2nd fraction.
7. Repeat step 6, discard the eluent.
8. Change the receiving plate. Wash the plate with 5 mL of MeOH. This is the 3rd fraction.
9. Repeat step 8 twice and collect the 4th and 5th fractions.
10. Wash the plate with THF/MeOH/TFA (1:1:0.01) 5x5 mL and dry. You can reuse the plate immediately.
11. Depending on your desired products, analyze either organic (1st and 2nd) or fluorous fractions (3rd-5th). Concentrate desired fractions and combine the products.

Notes:

1. For 48-well F-SPE, use 0.5 mL of DMF as loading solvent and cut the amount of washing solvent to one-third.
2. If your desired product is the non-fluorous component (using fluorous reagents or scavengers), step 6-8 can be skipped.

References:

1. Zhang, W.; Curran, D.P. "Synthetic applications of fluorous solid-phase extraction (F-SPE)" *Tetrahedron* **2006**, 62, 11837-11865.
2. Zhang, W.; Lu, Y.; Nagashima, T. "Plate-to-Plate Fluorous Solid-Phase Extraction for Solution-Phase Parallel Synthesis" *J. Comb. Chem.* **2005**, 7, 893–897.

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