



## 96-Well Gravity-Driven F-SPE Plate Catalog No. 807-0096

### General Information:

- Each well contains 0.75 g fluororous silica gel (125-210 $\mu$ m)
- Solvent volume up to 1mL
- Up to 50 mg crude sample loading
- Gravity elution

### DESCRIPTION:

The 96-Well Gravity-Driven F-SPE Plate<sup>1</sup> is developed for parallel separation of fluororous reaction mixtures which is complementary to the 24-well plate and automated F-SPE.<sup>2-4</sup>

**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:

#### Washing and Degassing of a New Plate:

1. Wash the fluororous silica gel with THF (1mL) followed by DMF (3 x 1mL).
2. Place the F-SPE plate into a 1L beaker or other container, submerging the whole plate in DMF.
3. Apply vacuum (<20 mmHg) for 5-10 minutes. Release vacuum and repeat this step.

**Note:** After degassing, the silica gel should look semi-transparent with no air bubbles.

#### F-SPE procedure:

1. Condition the F-SPE plate with your **wash solvent** (DMSO or 85:15 DMF/H<sub>2</sub>O) 3x1mL.

**Note:** If certain wells are extremely slow during conditioning, it is an indication of air bubbles in the well.

2. Add 0.5 mL of **washing solvent** to your sample. Sonicate or shake until a solution or suspension formed.
3. Load the sample solution or suspension onto the F-SPE plate. The eluent collected during loading can be discarded.

**Note:** The sample solution is often a suspension, so loading is best accomplished by either direct plate-to-plate transferring or using a multi-channel pipette. If an automated liquid handler is to be used, please ensure that it can accommodate suspensions.

4. Rinse your sample vials with 1 mL of **wash solvent**, load onto F-SPE plate and collect the eluent. This is the 1<sup>st</sup> fraction.
5. Change the receiving plate. (Make sure no droplet is left on the tips of the F-SPE plate.) Add 1 mL of **wash solvent** and collect the eluent. This is the 2<sup>nd</sup> fraction.
6. Repeat step 5 and collect the 3<sup>rd</sup> fraction.
7. Change the receiving plate. Wash the F-SPE plate with 1 mL of THF or MeOH. This is the 4<sup>th</sup> fraction.
8. Repeat step 7 three times and separately collect the 5<sup>th</sup> - 7<sup>th</sup> fractions.

**Note:** Do not permit the plate dry out during any THF (or MeOH) wash, otherwise, air bubbles will form and the degassing process will need to be performed.

9. Wash the plate with THF/MeOH/TFA (1:1:0.01) 5x1 mL followed by DMF 2x1 mL.
10. Depending on your desired products, analyze either organic (1<sup>st</sup>-3<sup>rd</sup>) or fluororous fractions (4<sup>th</sup>-7<sup>th</sup>). In most cases, the majority of non-fluororous compounds will be in the 1<sup>st</sup> and 2<sup>nd</sup> fractions and the majority of fluororous compounds will be in the 4<sup>th</sup>-6<sup>th</sup> fractions.
11. Concentrate desired fractions and combine the products.

#### Storage and Reuse:

1. For immediate reuse, follow the F-SPE steps described above.
2. For storage, condition the plate with DMSO 2x1 mL. Cap the plate, wrap it in aluminum foil then paper towel, and place in a zip bag for storage.
3. Before the next use, condition the plate with THF 2x1 mL, DMF 3x1 mL. Visually check the plate for air bubbles. If you see air bubbles, repeat the degassing procedure. Otherwise, the plate is ready for F-SPE.

#### References:

- 1) Zhang, W.; Lu, Y. *J. Comb. Chem.* **2007**, *9*, 836-843.
- 2) Zhang, W.; Lu, Y.; Nagashima, T. *J. Comb. Chem.* **2005**, *7*, 893-897.
- 3) Zhang, W.; Lu, Y. *J. Comb. Chem.* **2006**, *8*, 890-896.
- 4) Zhang, W.; Curran, D. P. *Tetrahedron* **2006**, *62*, 11837-11865.