



Automated F-SPE Using RapidTrace Workstation

General Information:

- The RapidTrace workstation is available from Caliper Life Sciences, www.caliperls.com.
- The software can control up to 10 units. Each unit uses 10 SPE cartridges charged with 2 g of fluororous silica gel (40-63 μm), and can purify ten samples sequentially if only one fraction (either non-fluorous or fluororous) is collected.
- Use 16x100 mm test tubes as collecting vessels and Eppendorf centrifuge tubes as sample vials.
- Up to 200 mg of sample can be purified. However, **100 mg or less is recommended**.

DESCRIPTION:

F-SPE using RapidTrace workstation is developed for automated purification of fluororous reaction mixtures. It is an element of the sets of F-SPE technologies¹⁻²

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](#)).

General procedure when collecting organic wash:

1. Add 0.5-1 mL of DMF to the sample and sonicate or shake until a solution or suspension formed. Transfer the sample to an Eppendorf centrifuge tube.
2. Wash the cartridge with 6 mL of THF (24 mL/min), followed by 6 mL of either DMF or MeOH (18 mL/min) depending on your elution solvent.
3. Condition the cartridge with 6 mL of **elution solvent** (8:2 MeOH/H₂O or 9:1 DMF/H₂O) (12 mL/min).
4. Load the sample solution or suspension onto the cartridge (12 mL/min).
5. Rinse your sample vials with 1 mL of **elution solvent**, load onto the cartridge (12 mL/min).
6. Elute the cartridge with 4 mL of **elution solvent** (12 mL/min).
Note: eluents from step 4-6 contains organic product and were collected in the same test tube.
7. Wash the cannula with 6 mL of THF twice, 6 mL of **elution solvent** once (24 mL/min)
8. Wash the cartridge with 6 mL of THF/MeOH/TFA (1:1:0.01) twice, MeOH 6 mL once (18 mL/min).
9. Analyze the solution in test tube and concentrate.

General procedure when collecting fluororous wash:

1. Conduct steps 1-7 described above. The eluents were not collected.
2. Elute the cartridge with 6 mL of MeOH (18 mL/min) and collected eluent contains fluororous product.
3. Wash the cartridge with THF/MeOH/TFA (1:1:0.01) 2x6 mL, MeOH 6 mL (18 mL/min).
4. Analyze the solution in test tubes and concentrate.

References:

1. Zhang, W.; Curran, D.P. "Synthetic applications of fluororous solid-phase extraction (F-SPE)" *Tetrahedron* **2006**, 62, 11837-11865.
2. Zhang, W.; Lu, Y. "Automation of Fluorous Solid-Phase Extraction for Parallel Synthesis" *J. Comb. Chem* **2006**, 8, 890-896.