Advances in Capillary Column Backflushing

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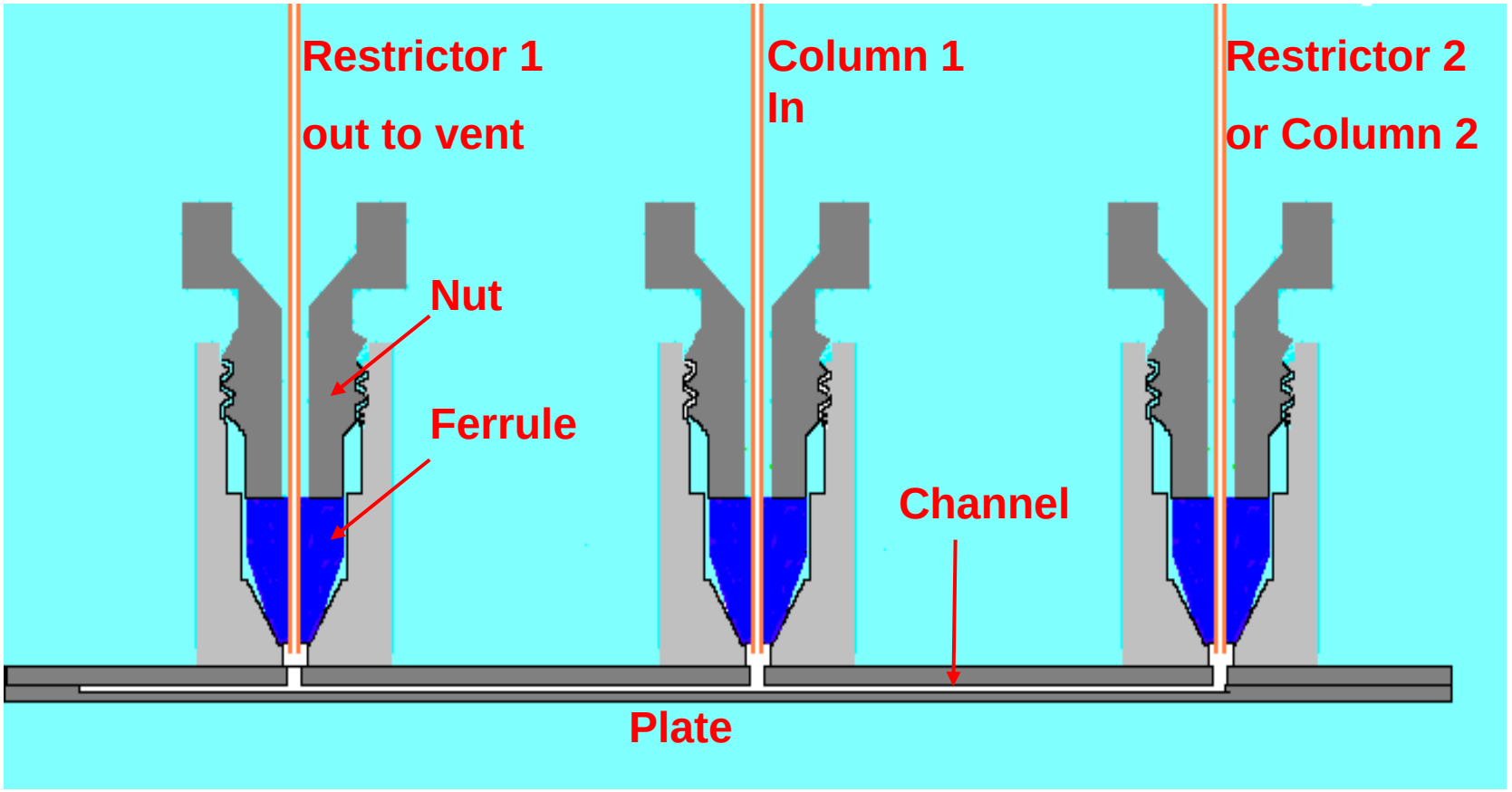
Capillary Flow Technology Traits

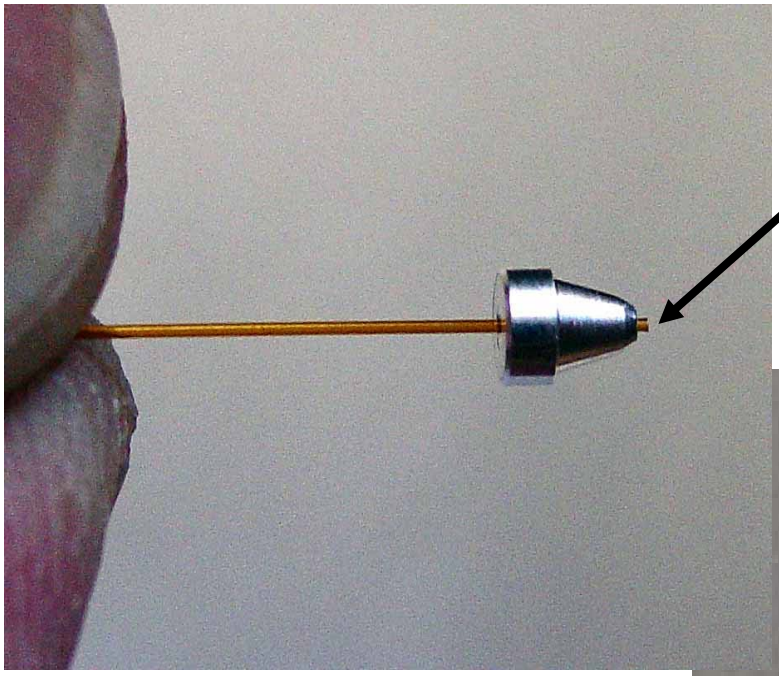
- Ease of use - ruggedness
- Inertness
- Usability to > 350 oC
- Excellent exclusion of air
- No unpurged volumes
- No porous materials into which compounds can dissolve ($\rightarrow$ peak tailing)
- Fast thermal response
- No outgassing or shedding of particles

FS Tubing Connections – The Magic

Unique design provides rugged, reliable connections with minimal unswept volume and high inertness

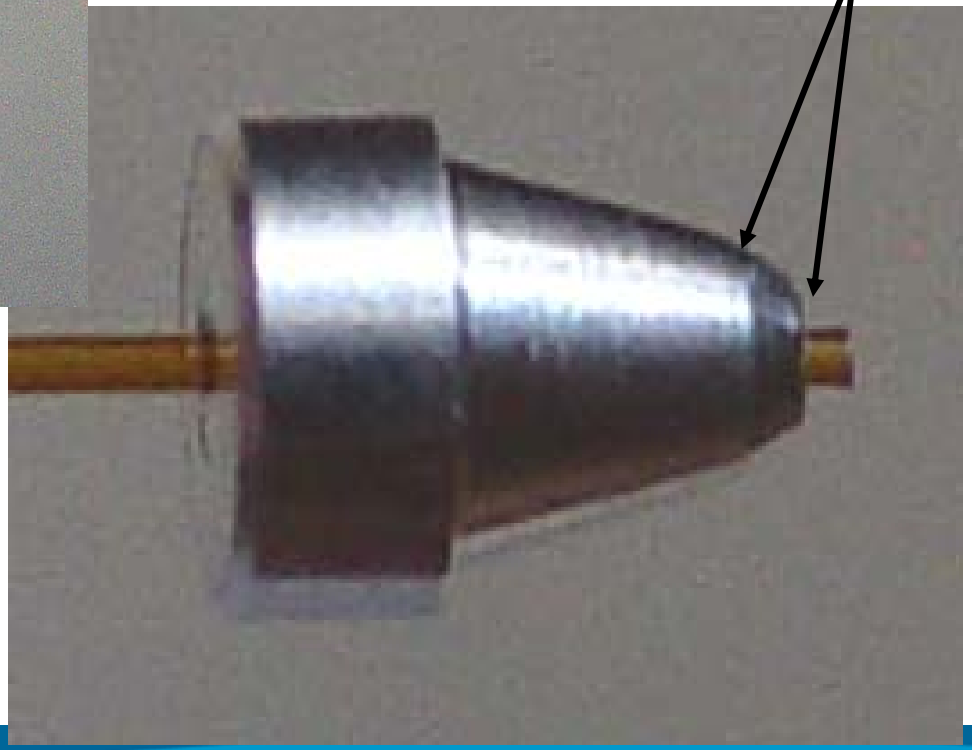


Ferrule Sealing



Square cut is not critical

Seal region



Backflush Setup

1. Start with working method and reference sample/chromatogram
2. Extract pertinent method information from current method
3. Select BF configuration
4. Prepare hardware (next slide)
5. Install columns
6. Determine pressure/flow settings, download and test stability
7. Screen standard on new configuration
8. Lock with migrated RTL calibration if RTL method
9. Pick last peak of interest using graphical tool, from which BF timing and conditions are recommended
10. Verify BF works
11. Fine tune BF conditions if necessary

Preparing hardware during setup

Assemble necessary HW & SW (e.g. PCM/EPC, CFT, Instrument Utilities SW)

Install any needed modules and accessory kits

Check frit (recommend “open/zero” frit)

Check/update PIDs (using instrument utilities SW)

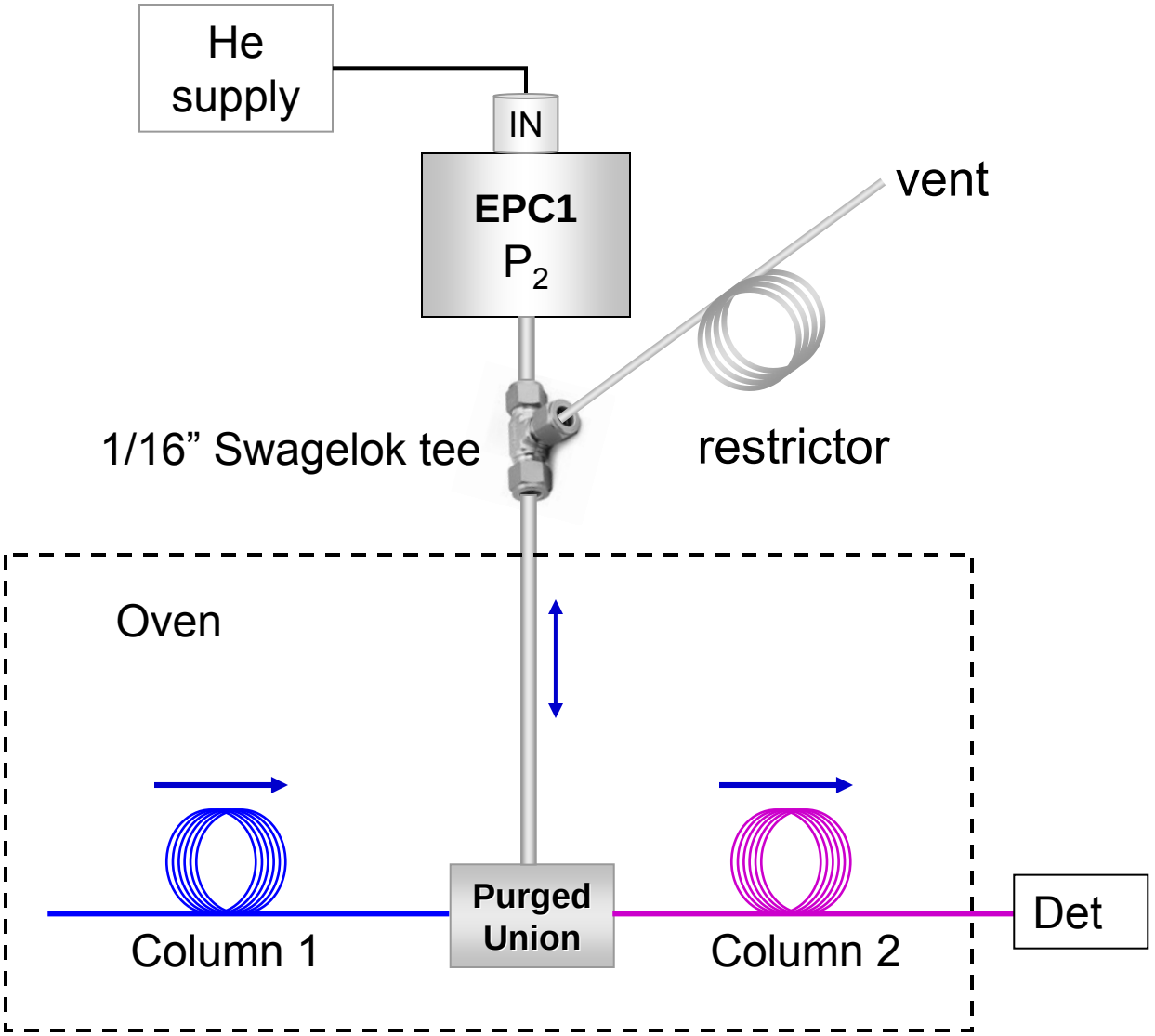
Zero all used pressure control zones (inlet, EPC)

Decide if you need a split purge and install preferred version

EPC/PCM instability at low ΔP or flow

- EPC and PCM modules were designed for most accurate and precise control at flows > 1 mL/min
  - Proportioning valve duty cycle should never be 0%
  - Symptoms are: long stabilization time, cycling P
- Some backflush configurations require flows of < 1 mL/min
- Adding a bleed ensures stable pressure control
  - Fixed bleed
  - Variable bleed

Basic Bleed Configuration



Purged 1/16” Union

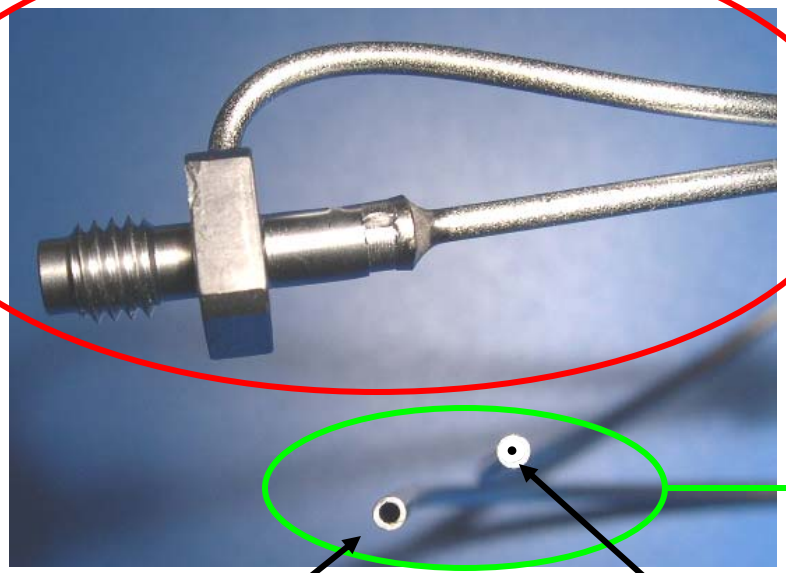
For purged splitting, in-oven connections

Eliminates air infiltration and stagnant lines

Provides bleed path for EPC stabilization and dumping excess column flow

Fixed restrictor configuration using G2855-80582

This is inside oven, replacing old Swagelok union



These are outside oven on top

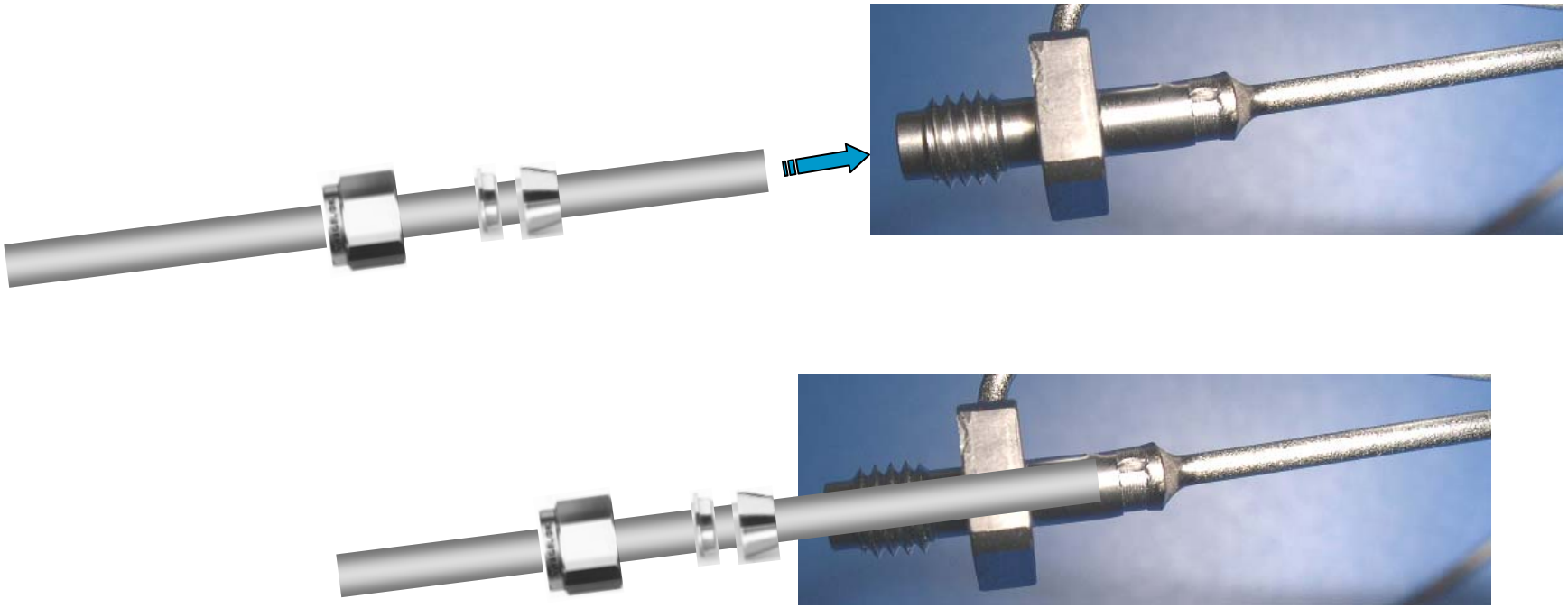
Big one goes to Aux EPC

Use 1/16” straight Swagelok union with Vespel ferrules

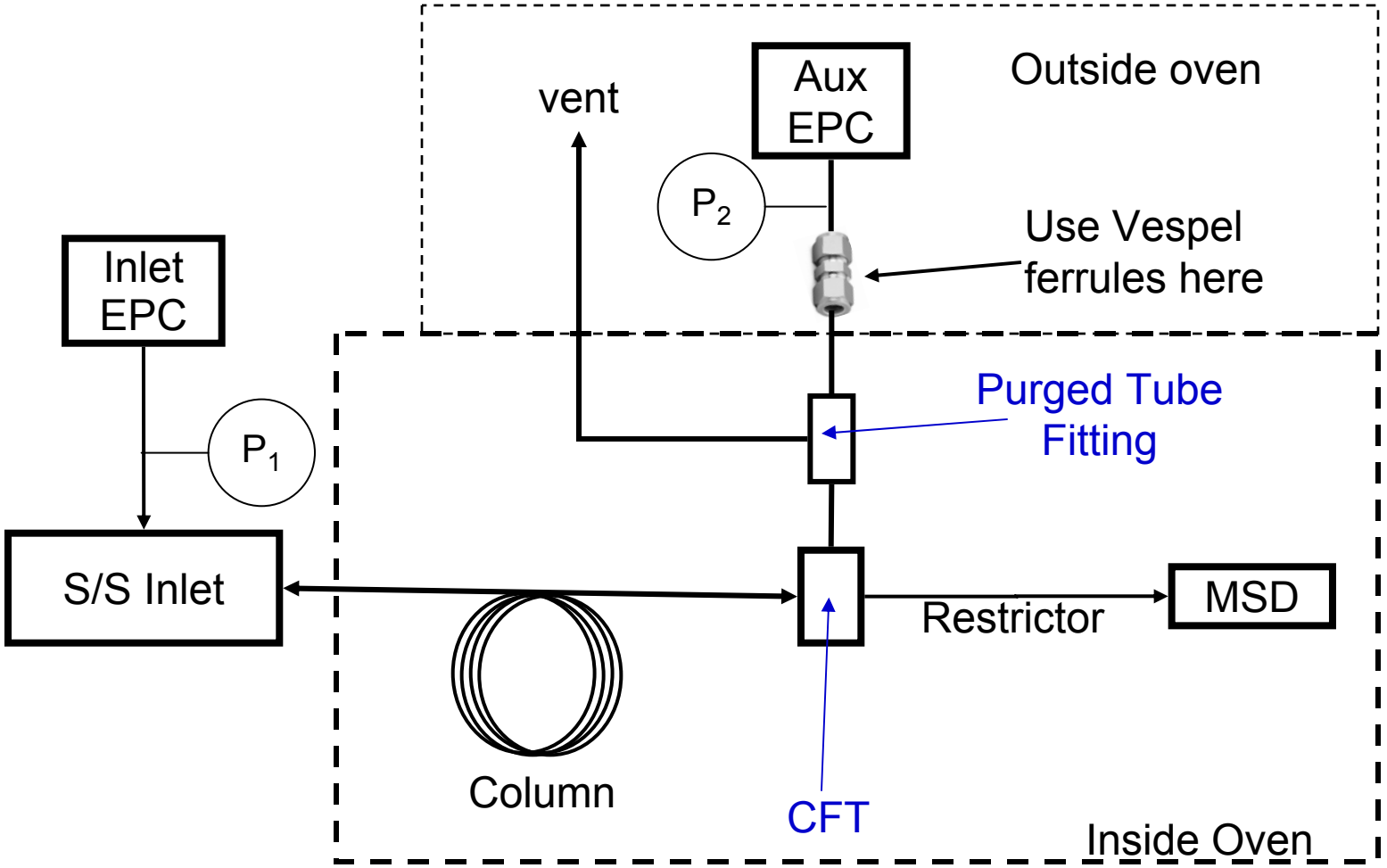
Small one goes to vent (room, vacuum vent, split vent trap, etc.)

Let hang loose or vent using whatever connectors necessary according to user requirements

Cut off and remake the QuickSwap in-oven fitting, with tubing extending fully into union (past the purge point)



Fixed split with purged tube fitting



Variable split bleed saves the day

Provides necessary bleed to allow pressure control solenoids to operate well (> 1 mL/min)

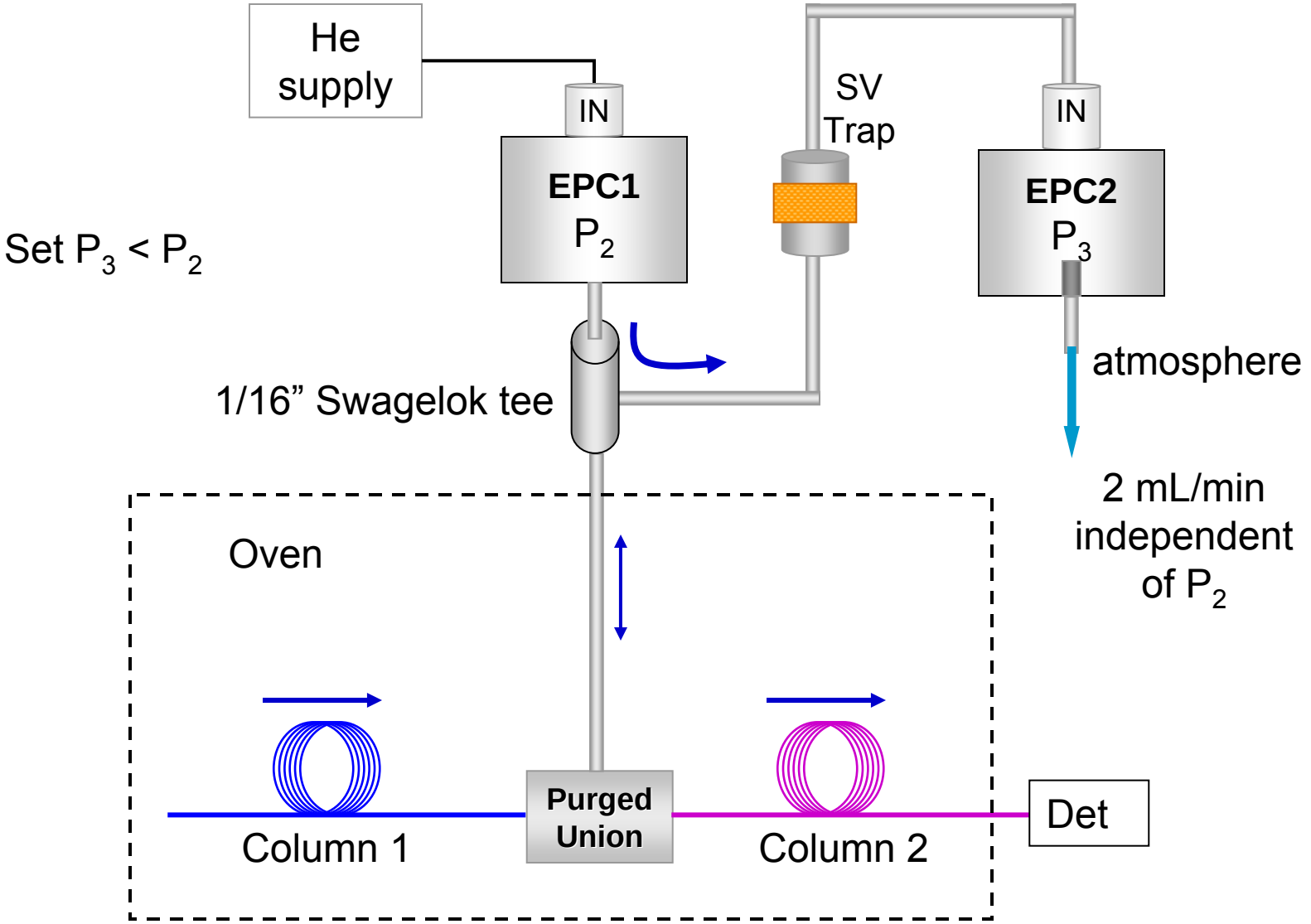
Maintains low flow even during high-pressure backflush

Uses normally unused pressure channel (EPC, Backpressure channel on PCM)

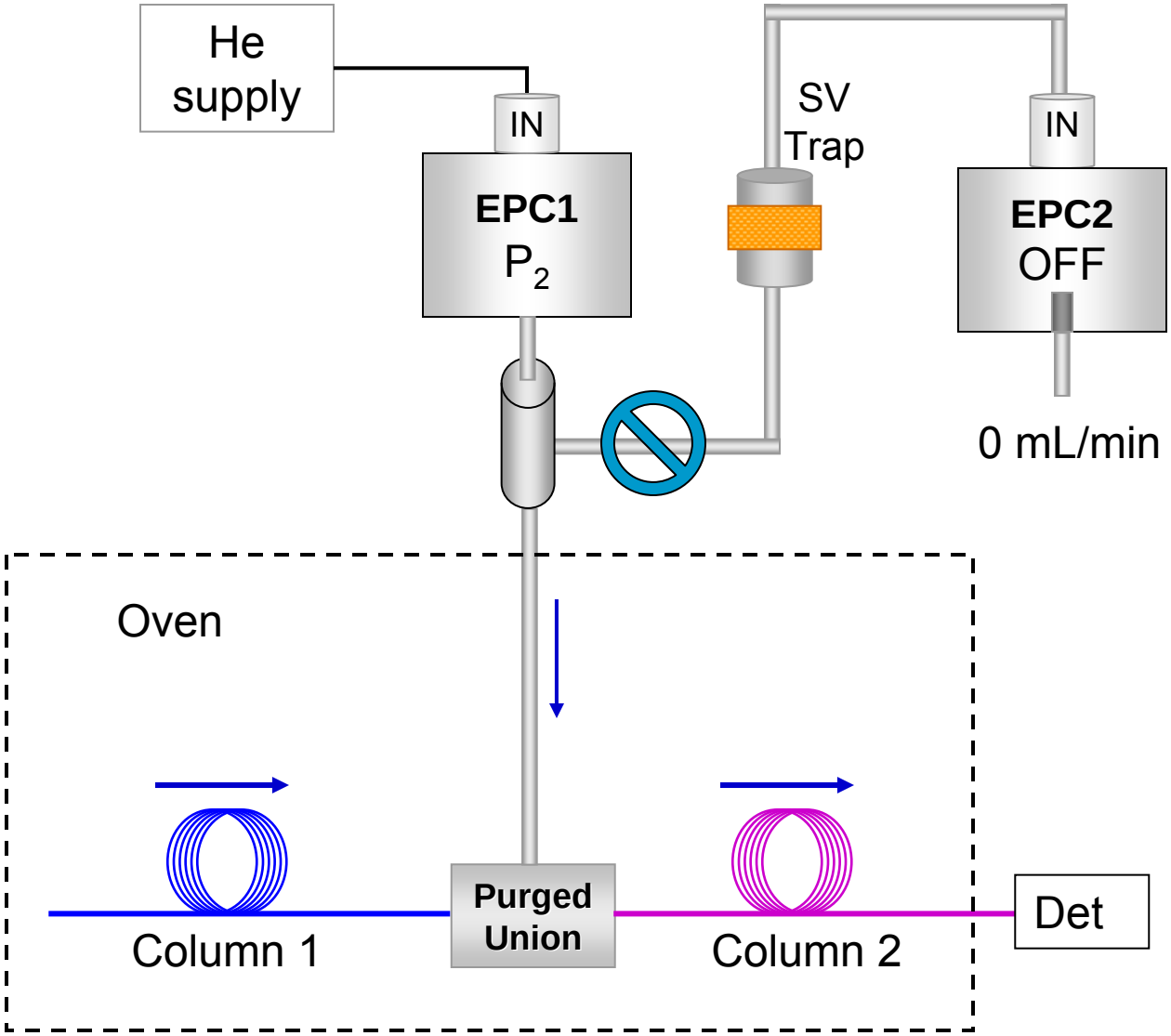
Allows split flow to be turned OFF for leak checking

Provides the opportunity to program bleed flow for custom applications

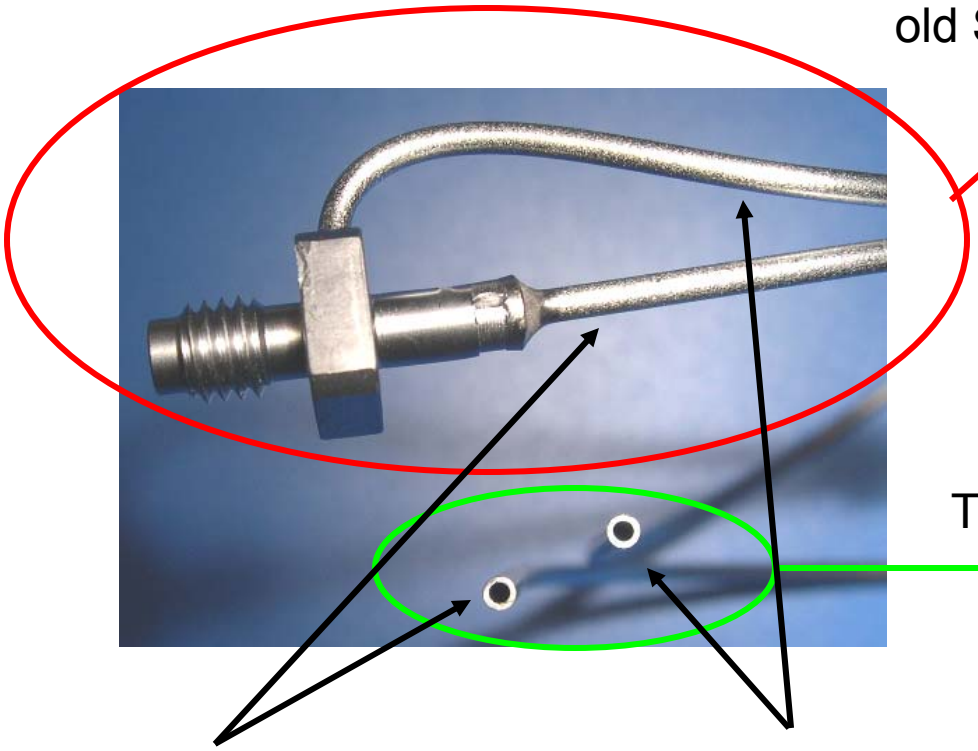
Using 2nd EPC for Bleed During Run



Bleed OFF During Leak Check



Variable restrictor configuration using G2855-80581



This is inside oven, replacing old Swagelok union

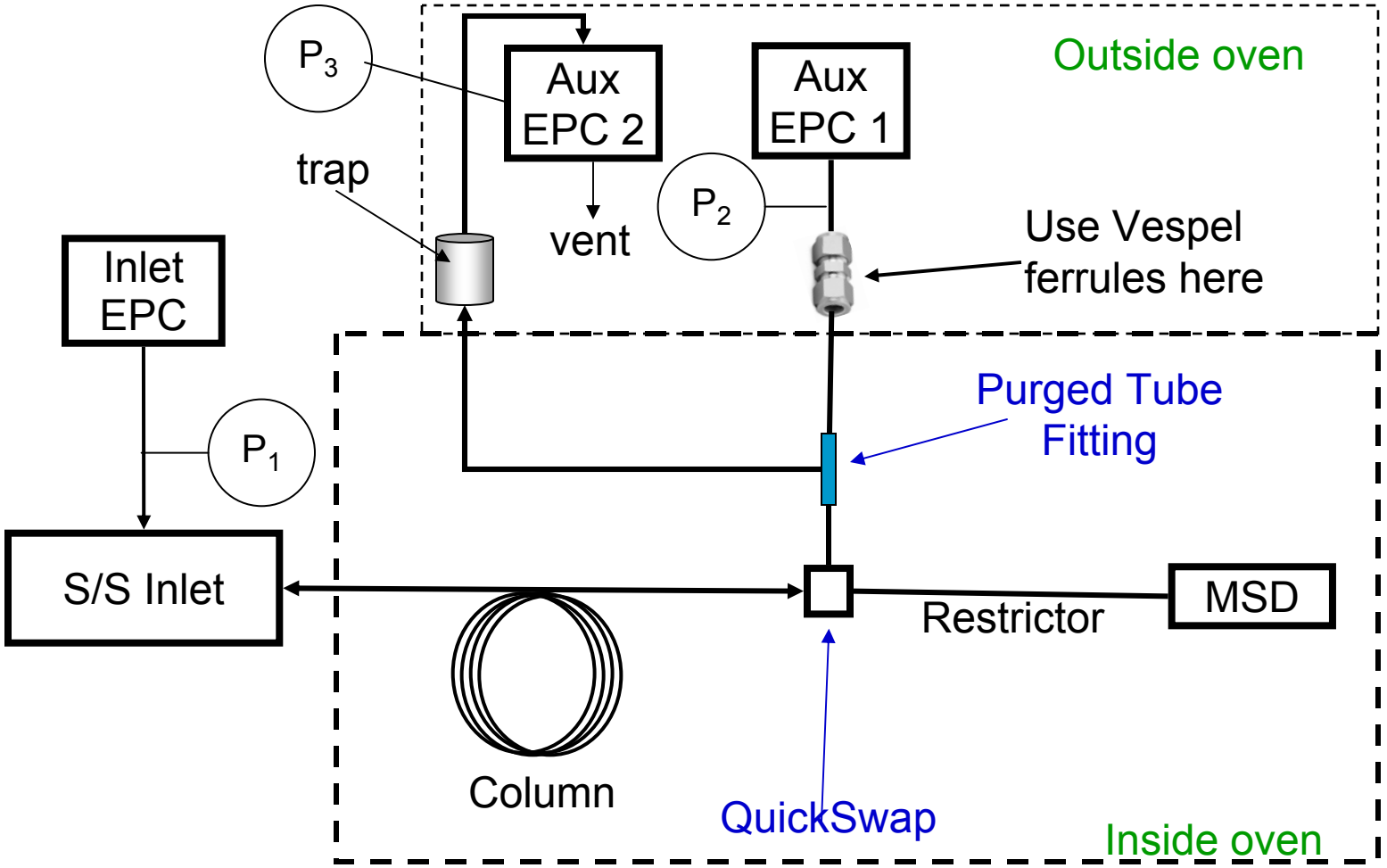
These are outside oven on top

From Aux EPC 1 (purge feed line)

Use 1/16” straight Swagelok union with Vespel ferrules

Vent to split vent trap then to input of EPC 2

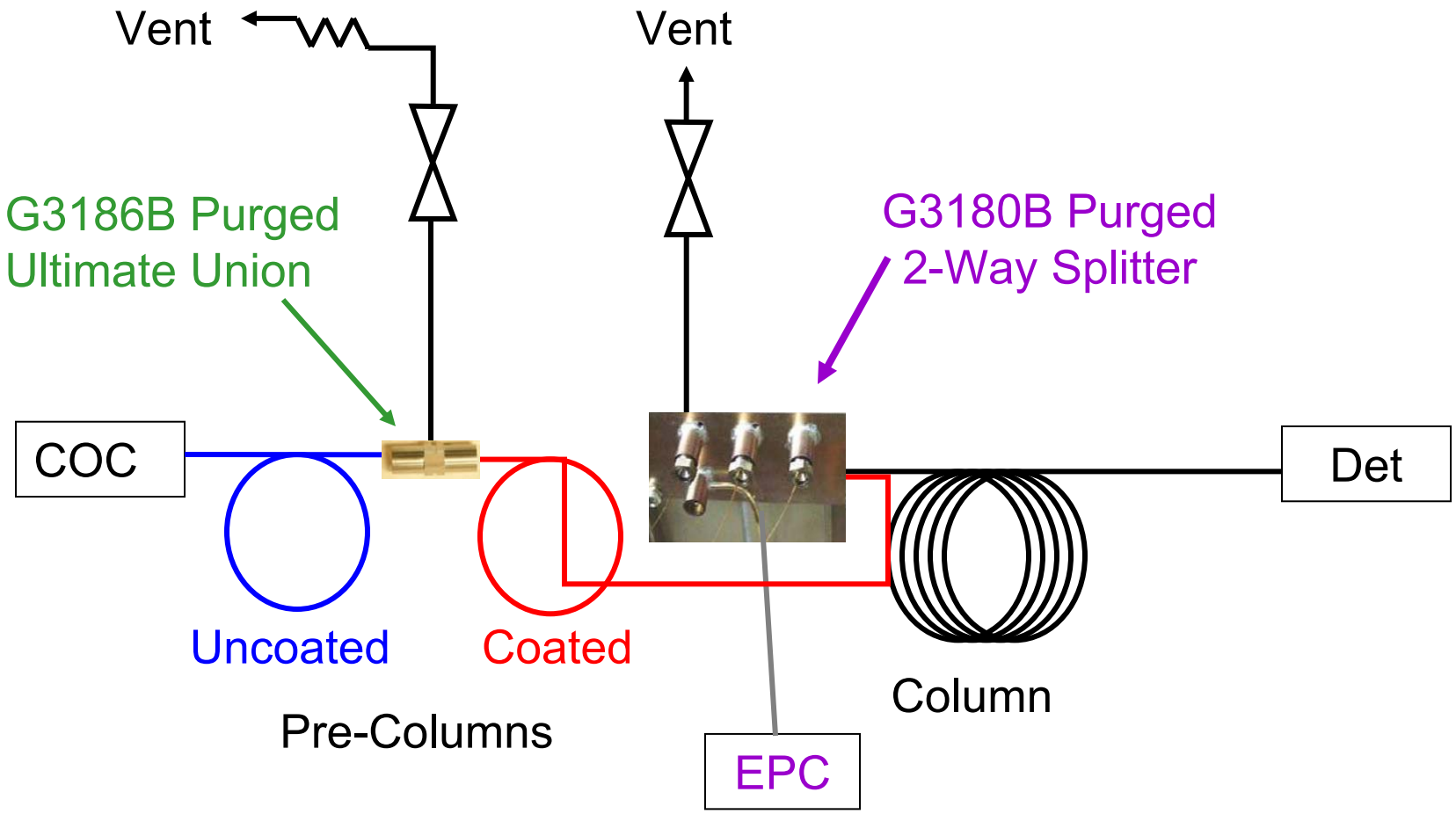
Post-Column BF with Variable Split



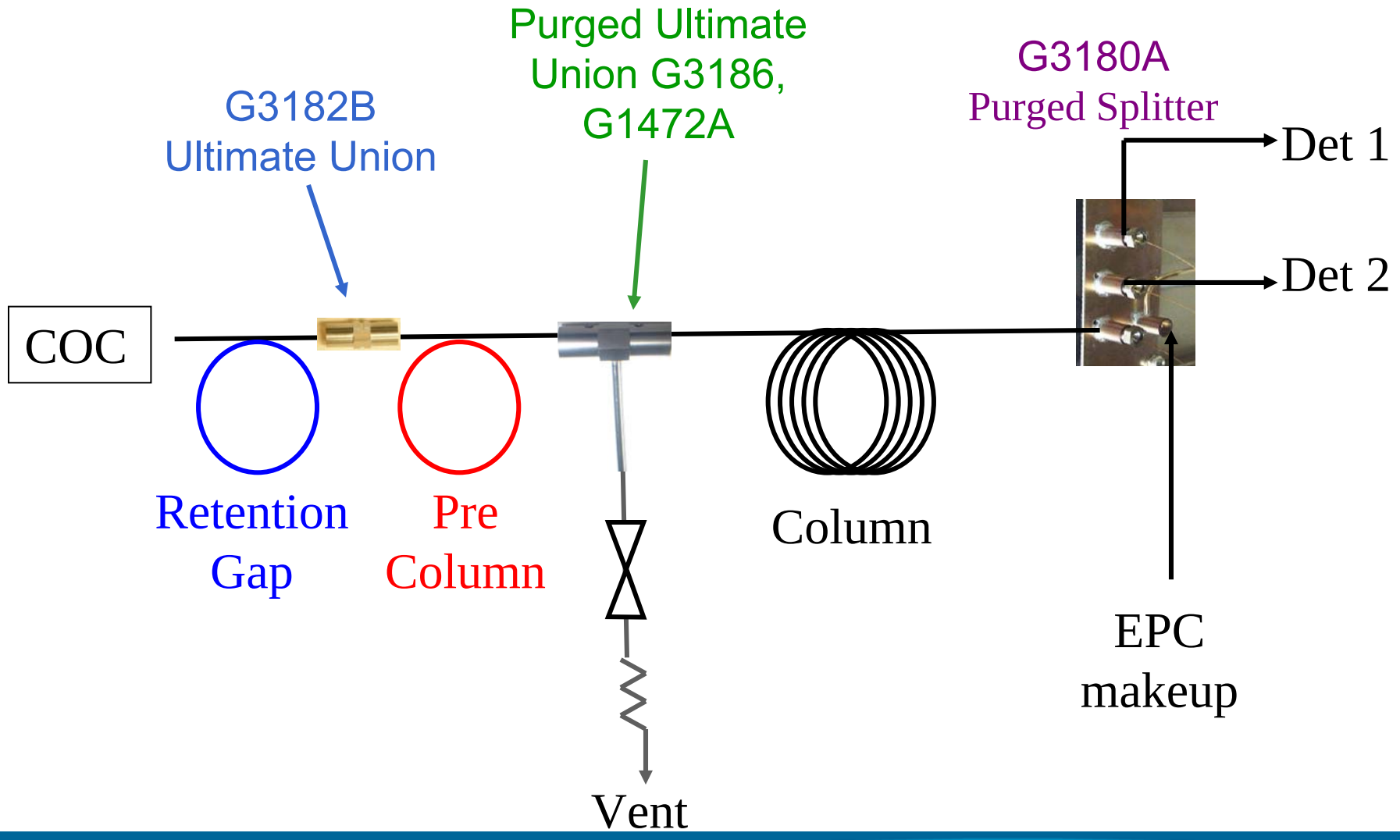
Large-Volume Injection, Cool On-Column Solvent Vapor Exit

Not for the novice!

LVI COC SVE with Pre-Column Backflush to Vent 1



LVI COC SVE with Backflush of the column to Vent 2



Conclusion

Capillary column backflushing has a large potential return – but requires some attention to detail to set up

Cost to implement backflushing is relatively low and is greatly facilitated by readily available instrument parts and accessories