



Injection methods in Flash purification - Dry-Load Injection



Dry-Load Injection

The dry-load injection is a convenient technique when the polarity of the reaction mixture or extraction is close to the polarity of the stationary phase or, when it contains solutes with extreme opposite polarity.

It should also be consider when:

- The compound of interest is retained more than the other compounds we want to separate.
- The sample contains one or several compounds having low solubility with the eluent.

Compared to liquid injection, the dry-load injection improves efficiency, resolution and final purity.

Sample in solution

Injection Loop



Limited injection volume due to sample solubility

SOLID SAMPLE

Dry injection



High pressure dry-load

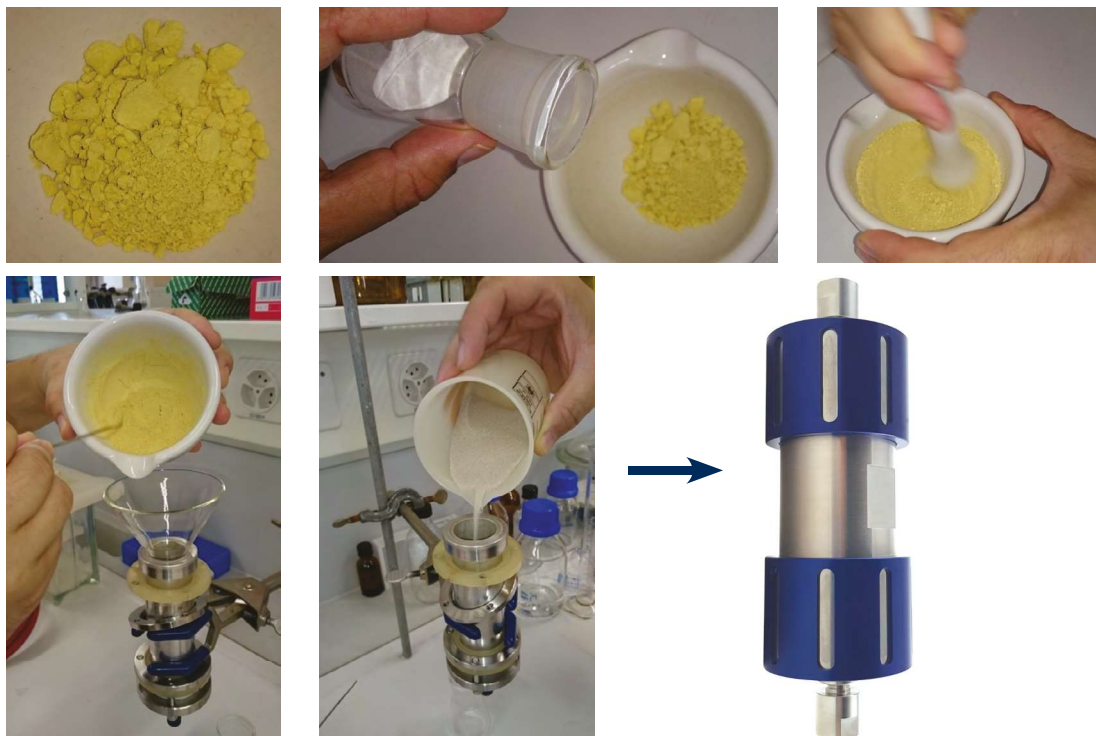


Allow to large sample amounts





How to prepare the Dry-load cell for injection



1. Adsorb the dissolved sample in the "better solvent" on a small amount of stationary phase (Silica, C18 or Celite).
2. Evaporate the solvent with a rotary evaporator until a "dry" powder is obtained. Note: If the volume of the dissolved sample is small, it can be poured onto the silica, and the partially impregnated silica mixed up to obtain a homogeneous dry powder, thus avoiding the passage to the rotary evaporator.
4. Place the mixture over the inlet frit of the column, once it has possibly been equilibrated with the elution solvent.
5. Add a sintered frit over the mixture, then a closure system or the piston of the column (for equipped systems) and slightly tamp the mixture to obtain a perfectly homogeneous deposit thickness.
6. Proceed with elution.

TECHNICAL TIP

The volume of the dryload must not exceed 5% to 10% of the purification column volume to keep sufficient resolution between fractions.

If possible, wet the dryload with 100% of the less eluent solvent before to start the purification run.

Caution: Adapt your step in function of the back pressure and the acceptable flow rate.

The dryload can generate air bubbles creating disruptions that hide the first peaks. (UV detector)

Celite exhibit advantages:

- It does not generate back pressure due to its large particle size
- It does not interact with the compounds that arrive at the same time at the top of the column
- It improves separation and is compatible with both NP and RP mode