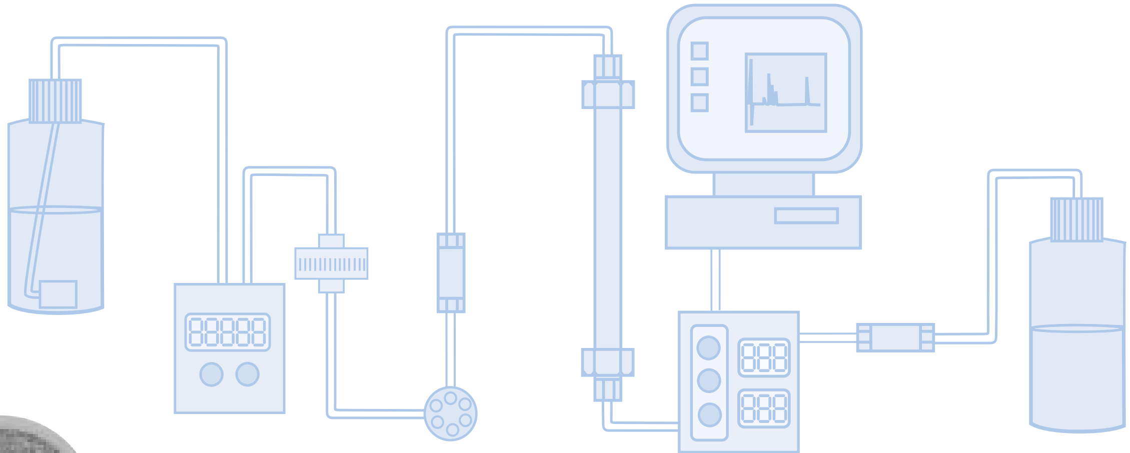
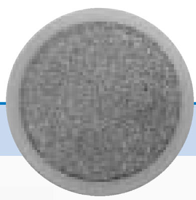
POROUS Metal Filter Products and the LC System





Frits for Chromatography

Frits and filters are porous metal products used in virtually all liquid chromatographic (LC) systems. Their function is to prevent unwanted particles from entering the LC system. These particles may come from the sample, the solvent or debris generated by the LC system itself (i.e. pump or injector). Particles entering the LC system may lead to clogging of capillaries, interference with the chromatography by changing chromatographic parameters or disturbance of the detector function.

The most important characteristic of a frit, besides the diameter and the thickness, is the porosity. When considering porosity, it is not only the average size of the pore that is of interest, but also the size distribution and the amount of pores available. The size distribution should be a gaussian function with narrow tolerances. At Applied Porous Technologies, we continuously monitor our manufacturing process and maintain strict control of the powders used in our process in order to provide the tightest pore distribution possible.

In addition to pore distribution, it is important to consider the pore density, or number of pores available. Take, for example, a frit with 2 µm porosity and a surface of 0.25". The theoretical maximum amount of pores with 2 µm average diameter would be about 5,000,000. This frit would give you the highest possible flow achievable. Based on standard bubble point methodology, the same frit having just a few pores would also qualify as a 2 µm frit. Although the porosity may be within specification, it is unlikely that that this frit would provide adequate flow. Careful control of our powders and process allows Applied Porous to offer the best pore size distribution while maximizing flow properties.

At Applied Porous Technologies, we understand the needs of the chromatography industry. We work closely with many leading instrument manufacturers and column packing companies to provide the best products available. Our employees are committed to continuously upgrading our products through new product development and process improvement. We are always open to discussions and ideas on how to make our products and services better.

The Liquid Chromatographic System

The first and most obvious filter you will see in a liquid chromatographic (LC) system is the ***solvent inlet filter***. This is located in the solvent reservoir and prevents dust particles, general debris and, to some extent, bacteria from entering the LC system. These contaminants can interfere with downstream in the system such as the low-pressure gradient former or the pump. Particles entering the valves of either of these two instruments may interfere with the proper function. The result could cause an increased baseline noise, nonrepeatable gradient forming, unreliable flow rate or other interferences with the LC system. In general, the solvent filter is a low-pressure filter and will allow a high flow rate due to a large surface area and large porosity. The high flow rate is necessary to guarantee that no extra force is needed for suction of the solvent from the solvent reservoir to the pump. It is also often recommended to have the solvent level slightly higher than the pump, helping to create a slight prepressure on the pump head and valves.

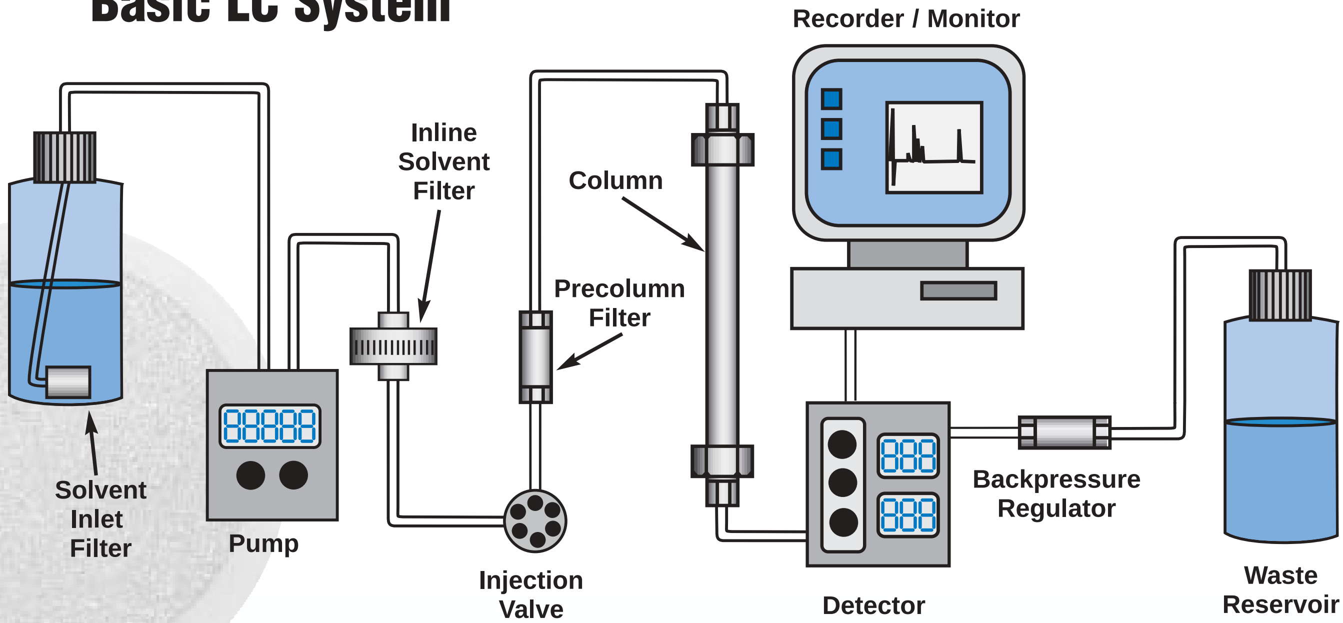
All moving parts within the LC system can generate debris. Abrasion from the ***pump*** piston seals is one of the most common sources. Despite the superior sealing materials available today, small irregularities in the seal itself or on the piston, dirt on the piston or an improperly installed seal will result in small particles being removed from the seal and washed downstream towards the ***injection valve***. Regardless of whether your system has a manual or automatic injection system, all injection valves have close tolerances (flat surfaces moving against each other to provide sealing under high-pressure conditions). Improper valve operation can occur as a result of debris interfering with proper sealing of the valve. Alternately, debris entering the valve can destroy the sealing surfaces, generating additional particles and making it necessary to repair the valve or replace the rotor seal. To prevent this costly damage, a large-surface, high-porosity ***inline solvent filter*** is placed in line between the pump and the injection valve. These solvent filters have easily replaceable filter discs that can be changed out for a fraction of the cost of repairing a damaged injection valve.

Another source of particles in the system is from the ***sample*** itself. Whenever possible, the sample should be prefiltered prior to injection into the system. Once injected, the sample travels through capillary tubing towards the column. Particles entering with the sample, or those generated by the injection valve, can easily clog the capillary. Debris passing through the capillary will collect in the column and can also affect the performance. To prevent these problems, it is recommended that a low-porosity, small surface area (hence small dead volume) ***precolumn filter*** be added between the injection valve and the column. Again, the cost to replace the filter element in the precolumn filter is minimal in comparison to the cost of the column or the time lost to replace plugged capillary tubing.

The ***column*** is the one part of the LC system that always uses porous frits. A typical column will have two frits, one at the inlet and one at the outlet. The frit on top of the column, or inlet, prevents particles from entering the bed of the column. This frit has a protective function. Any debris that enters the column will be trapped on the inlet filter. Even though the frit can eventually become clogged, the expensive column bed will remain intact.

The inlet frit also aids in the distribution of the solvent/sample over the column. A column with 4.6mm I.D. has a surface area that is 330 times (0.25mm I.D.) or 1000 times (0.13mm I.D.) larger than that of the capillary. The solvent stream has to be distributed evenly over the column surface to give the best results for the separation. The same is true for the outlet frit, where the solvent stream has to be concentrated from 4.6mm into the small capillary I.D. without band broadening. The ideal frit for the inlet would have to have a larger porosity and minimal

Schematic: Basic LC System



thickness in comparison to the outlet frit. This would minimize pressure losses and reduce the amount of dead volume. Often, however, the same frit size is used on both sides of the column to prevent the user from reversing the column and switching the flow direction.

The primary function of the outlet frit is to retain the packing material. Here it is important to use a frit with a porosity smaller than the packing material (i.e. a 5 µm packing material would need a 2 µm frit). Care must be taken not to choose too small of a porosity for the outlet frit. All packing material contains some smaller particles and through attrition will also break into smaller particles. If you choose your frit with a very small porosity, the small particles contained or generated in your packing material will eventually work into the pores and clog the frit, resulting in increased backpressure. It is generally safe to choose a frit for your column outlet with a porosity about half the size of your packing material.

After the sample has left the column it will enter the ***detector***. It is desirable to keep the void volume between the column outlet and the detector cell as short as possible. Small particles of the packing material can penetrate the outlet frit and will then enter the detector. The use of a large-volume filtering device between the column outlet and the detector can result in band broadening. At the same time, the detector cell has to be protected from column packing material particles. A thin frit with small pores and small surface area might work well here to filter out particles while still keeping void volume low. This will not work for all separations and is very unusual to find in practical work.

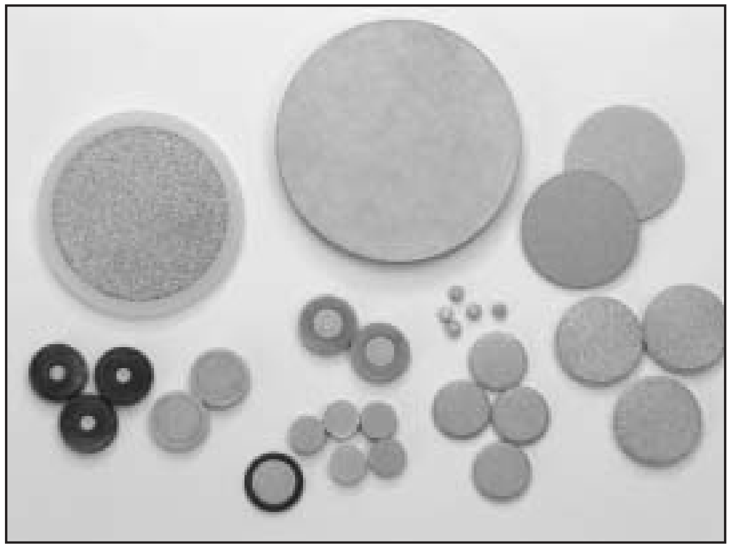
Due to pressure differences and the relaxation of the solvent, small gas bubbles can form. The formation of bubbles can be prevented by the use of a ***backpressure regulator***. A porous metal frit in an in-line filter can be used as a backpressure regulator, but care must be taken since a clogged frit will continuously increase the backpressure in the detector. Most detector cells are not designed for high-pressure applications, therefore we recommend the use of a standard backpressure regulator.

Expert Advice: Choosing the Right Frit

There are a number of things to consider when deciding on what type of frits and filters to use in an LC system. The choice of material is very critical to the function of the frit. Our standard material is 316LSS, which is suitable for many applications. We also offer our porous frits in Titanium, Hasteloy and a number of other materials for applications that require greater corrosion resistance or biocompatibility.

Many frits are also supplied with a press-fitted polymer ring. This ring has two functions. First, since a stainless steel frit will not seal well against a stainless fitting, the ring acts as a gasket. Additionally, the ring fills up a void in the fitting thereby reducing dead volume. Applied Porous offers frits with rings in a wide variety of polymers including PTFE, ETFE, PEEK and Kel-F. The end user must choose the combination of porous metal and polymer that will perform the best with the intended sample and buffer chemistry.

Frit geometry is another consideration. Frits can be produced with square or chamfered edges, depending on the preference of the end user. The chamfered edge facilitates assembly in applications where the frit is pressed into another component. Some column builders, however, prefer the straight-edged frits because they minimize dead volume. For more demanding applications, we offer a variety of specialty frits such as a dual-density frit with a 5 µm porosity in the center and 1µm around the periphery. For preparative columns, we manufacture a multi-porosity frit that has a coarse support layer mated to a very thin fine filtration layer. This combination provides excellent filtration with minimal pressure drop. Frits are also available with embossed star patterns for distribution, special chamfer designs, or assembled into custom fittings and hardware. One of the more unique frits manufactured was parabolical. In tests at the Technical University of Berlin, this frit was shown to provide extremely uniform flow distribution in a preparative column. It is this type of effort that will allow us to produce the next generation of filter products for chromatography.



Chromatography Frits and Polymer Ring Assemblies

Porous Metal Products for the chromatography industry are only a small part of what we do at Applied Porous Technologies, Inc. However, our dedication to offering the widest variety of options to our chromatography customers is just one example of our commitment to be the number one supplier of porous metal products in the industry. Working alongside our customers, we are constantly developing new and innovative solutions. The experience and knowledge of our employees, combined with the rigid control of our manufacturing processes, strict quality control and wide variety of available base materials, guarantees that we can supply the right solutions for your chromatography applications.

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