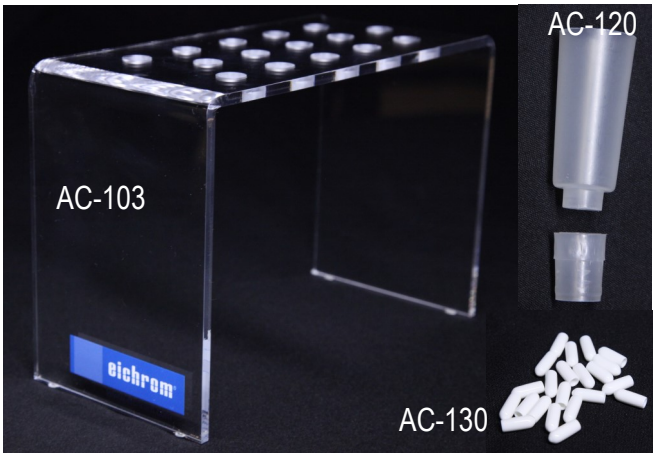
AN-1703-11

# Packing Eichrom 2 mL Columns

**Summary** Eichrom offers empty 2 mL columns and bulk resin for customers who wish to pack their own columns. This application note will offer advice on slurry packing columns that will exhibit favorable flow conditions and efficient separations. Some hydrophobic, difficult to wet resins may require additional treatment prior to slurry packing or may be dry packed.

Eichrom snap tip 2 mL Columns (AC-141-AL) come with the bottom frit inserted. Top frits and column top caps are also included.




2 mL Column Racks (AC-103), Column Tip Closures (AC-130), and 2-piece 25 mL Extension Funnels (AC-120) are also available.

The first step in slurry packing columns is to wet the resin by mixing with an appropriate aqueous phase. For most Eichrom resins, a solution of 0.05M $HNO_3$ is ideal for column packing. Add a volume of resin sufficient to pack your columns to 3-5x that volume of 0.05M $HNO_3$. Mix the resin by vortexing, swirling or gently tumbling. Avoid vigorous shaking, which can lead to air bubbles that can degrade column flow and separation efficiency. If a portion of the resin floats on the surface of the 0.05M $HNO_3$, centrifuge the sample. Repeat mixing and centrifuging as necessary to achieve a well wetted resin with minimal amount of floating material.

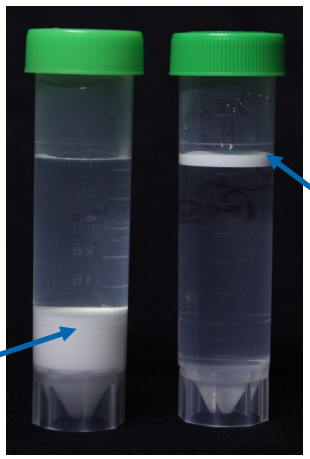
Some more **hydrophobic resins will not wet well in 0.05M $HNO_3$**. Table 1 lists some difficult to wet resins and **alternative matrices to facilitate wetting**. The resins in Table 1 can be wetted by **replacing the 0.05M $HNO_3$ with the alternative slurry matrix** and following the steps above. Once DGA, LN2 and LN3 have been wetted with the alternative slurry matrix, centrifuge and decant the aqueous phase and replace with 0.05M $HNO_3$ for storage and column packing. Higher concentrations of acid may enable faster wetting, but may be more dense than the resin. Once wetted, the high acid concentrations can be remove or diluted to allow resin to sink.

**Table 1. Slurry Matrices for Difficult to Wet Resins**

| Resin | Slurry Matrix |
|---|---|
| Prefilter | 0.05M $HNO_3$ |
| Ni Resin | 0.15M Ammonium Citrate |
| DGA, Normal | 2-4M $HNO_3$ |
| DGA, Branched | 2-4M $HNO_3$ |
| Cu Resin* | 0.05 - 2M HCl |
| LN2 | 1M $HNO_3$ |
| LN3 | 2M $HNO_3$ |

*Cu Resin will float on the surface of the solution even when wetted.

Wetted resin, ideal for slurry packing columns.



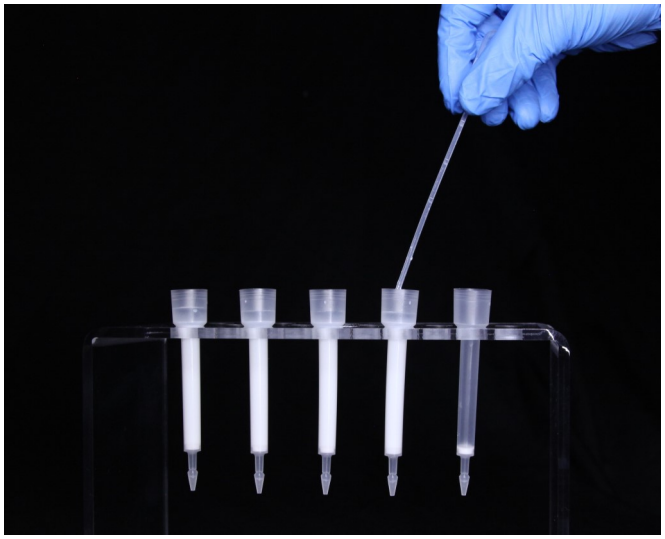
Floating or poorly wetted resin, difficult to slurry pack into columns.

The packing method is written assuming a 0.05M $HNO_3$ slurry matrix. For Ni Resin and Cu Resin, replace 0.05M $HNO_3$ with the appropriate alternative. Pre-packed Eichrom 2 mL columns contain 1.6 mL of resin. This method was written to replicate this fill volume.

As the resin is wetting in the appropriate matrix, add empty 2 mL columns to a column rack or other support. Add a small volume of 0.05M $HNO_3$ to soak the bottom frit and remove air bubbles. Soak until no can be seen escaping from the frits.

Add enough top frits for each column to a centrifuge tube with a small volume of 0.05M $HNO_3$. Soak the frits to remove air bubbles. Swirl or vortex to mix, but avoid vigorous shaking.

Decant the 0.05M $HNO_3$ from the empty columns. Mix the slurry of resin and 0.05M $HNO_3$ to suspend the resin. Add the resin slurry to each column until the reservoir above the column is ~half full. Allow the resin to settle (~1 hr).



Full 2 mL columns should have a bed height of 4.1 ± 0.2 cm. Add additional slurry to meet this height or remove excess resin using a plastic transfer pipet. Leave enough 0.05M $HNO_3$ above the packed bed to fill the column and a portion of the reservoir.

Place a pre-soaked frit into each column. Using a glass or plastic stir rod, push the frit to the top of the packed bed. Decant the 0.05M $HNO_3$ above the top frit and rinse away any residual resin from above the top frit using 0.05M $HNO_3$.

If storing the columns for future use, fill the reservoir above the top frit ~half full with 0.05M $HNO_3$ and place a top cap on each column. If using the column immediately, snap off the bottom tip, allow any excess 0.05M $HNO_3$ to drain, and begin the column preconditioning step.

**Dry Packing Columns** Some difficult to wet resins can also be dry-packed into columns:

1) Place 2 mL columns with bottom frits in column rack.
2) Weigh 0.65 ± 0.05 g of dry resin into each column.
3) Tap to settle the resin.
4) Place a top frit on each column and push the frit to the top of the resin bed.
5) Rinse away any excess resin above the top frit.
6) Add preconditioning solution to the column reservoir. Over pressure or vacuum may be required to initiate column flow.
7) Allow solution to drain through column.