0.2 ml RE-Spec (50-100 mesh) Columns for Whole Rock Bulk REE Separations

Column Procedure:

1. clean resin 3 ml 0.05 N HNO₃ 2. precondition column 3 ml 2 N HNO₃

3. load sample dissolved in 0.2 ml 2 N HNO₃

 4. wash
 0.1 ml 2 N HNO3

 5. wash
 0.1 ml 2 N HNO3

 6. wash
 0.1 ml 2 N HNO3

 7. wash
 0.5 ml 2 N HNO3

 8. wash
 0.5 ml 2 N HNO3

 9. wash
 1.0 ml 2 N HNO3

 10. wash
 1.5 ml 2 N HNO3

11. elute REE 4 ml warm* 0.05 N HNO₃

Resin Cleaning:

RE-Spec resin is a rare earth element selective extraction chromatographic material produced by EIChroM Industries, Darien, IL. We preclean the resin by placing an aliquot in a small Teflon bottle which is then filled with quartz-distilled water, shaken well, and placed on a hotplate set at around 50°C overnight. The next day the water is decanted and the procedure is repeated ten times. Once the resin is ready for use, it is transferred to a dropper bottle where it is dispensed as needed. The resin in the columns is disposed of after every use.

For details on this resin, see:

Horwitz, E.P., Chiarizia, R., Dietz, M.L., and Diamond, H., 1993. Separation and preconcentration of actinides from acidic media by extraction chromatography. Anal. Chim. Acta, 281: 361-372. (note: this paper discusses TRU-Spec, an analogous extraction chromatographic material to RE-Spec, but with a lower ratio of CMPO/TBP)

Column Construction and Cleaning:

Start with 5.5 cm KORVEX $4 \times \frac{1}{2}$ shrink tubing. Fit the shrink tubing over the end of a standard test tube with approximately 2.5 cm of the tubing extending beyond the end of the test tube. Shrink this portion of the tubing with a hot air gun as far as it will go. This will result in a column with an interior diameter of around 3.6 mm. Place a polystyrene frit in the end of the column and adjust it so that the volume of the column is 0.2 ml. The reservoir (the unshrunk portion of the column) has a volume of 3 ml.

After use, the columns are placed in a Teflon bottle which is then filled with clean water and placed on a hotplate at a low setting for a couple of days. After this, the water is decanted, the columns are rinsed, and 7 N HNO₃ is added to the bottle and placed back on the hotplate at a low setting for a couple more days. After this, the HNO₃ is decanted and the columns are rinsed with clean water and are ready for use.

Questions? Contact Todd Housh (housh@mail.utexas.edu)

^{*} use of warm acid helps overcome a kinetic effect which results in severe tailing of the REE when room temperature acid is used.