

# Lithium Isotope Sample Preparation

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overview This is a set of procedures for processing samples for Li isotope analysis in the Clean Lab (CHEM 0216). It is required that anyone undertaking these procedures undergo Personnel Safety Training. Two sets of column procedures are included, one based (**12 ml column chemistry**) on the technique of Tomascak et al. (1999) and another (**three-column chemistry**) based on the technique of Moriguti and Nakamura (1998).

reagents For cleaning Teflonware, reagent grade acids are used. These are stored in the respective areas under vented laminar flow cabinet #5. Most acids for sample digestion and chemistry are single-pass quartz distilled (1x QD). The concentrated stock of these are adjacent the reagent acids under cabinet #5. **Do not use anything but acids clearly labeled 1x QD.** Higher purity acids are precious and in heavy demand for other techniques. Concentrated acids are, in general: HCl ~12 M, HNO<sub>3</sub> ~ 15 M, HF ~ 49 M. In this text and in the lab acids are of two general types: concentration-calibrated (labeled with concentrations, to two to three significant figures), and concentration-approximate (for example, “~6 M HCl,” meaning it has been mixed to this approximate concentration, but not calibrated). The latter acids are good for either using in column cleaning or for later dilution and calibration. Do not use concentration-calibrated acids for cleaning.

Concentrated HCl and HNO<sub>3</sub> are dispensed to dropper bottles on the lazy Susan in vented laminar flow cabinet #4, where the digestions take place. Also on the lazy Susan are a squirt bottle with Seastar concentrated HF and a Teflon vial containing Seastar concentrated HClO<sub>4</sub> (perchloric acid). Seastar is a commercial manufacturer of high purity reagents. The organic solvents used in the chemistries are methanol and ethanol, both ~100%, stored under cabinet #4, in the area marked “organics.” It is necessary for the 12 ml Li column chemistry to use Teflon-distilled methanol. The ethanol that is used in the three column chemistry is HPLC-grade, not additionally distilled.

Reagents commonly used in the Li column chemistry are kept in the workstation with the primary columns or in the front workstation with the REE columns. Most of these are dedicated to Li chemistry and bear labels indicating this status. A polyethylene 500 ml squirt bottle of 1x QD ~6 M HCl is kept on the shelf with the Sr-REE chemistry reagents in the rear left of the lab.

Additionally, 500 ml squirt bottles containing Milli-Q water (MQ H<sub>2</sub>O) are dispersed throughout the lab. These should be refilled after use, not left empty. The reservoir to the Milli-Q system should be left full after use (via the red valve to the right of the system). A squirt bottle with ethanol used for removing ink from surfaces is ordinarily kept near the Milli-Q system.

cleaning Teflonware Digest samples in 15 ml (or 7 ml) screw-top Teflon (Savillex PFA) vials and use 180 ml (or 120 ml) screw-top Teflon jars for collecting Li in the cation exchange separation chemistry. There are sufficiently few jars that they must be cleaned immediately after samples are out of them. The cleaning process can be rushed so as to take as little as a few hours (if time is crucial), but otherwise will take two nights (or longer if items are not imminently needed).

1-remove labels with ethanol from a squirt bottle and rinse interior and exterior with tap H<sub>2</sub>O and swab interior surfaces with a moistened Kimwipe—**never** use a hard, abrasive, or sharp object (e.g., fingernail, scrub brush) on Teflon PFA! This variety of Teflon is soft and easily scratched. Scratches are loci for difficult-to-extirpate material that can easily cause long-term blank problems.

2-rinse with MQ H<sub>2</sub>O squirt bottle

3-add ~6 M HCl (50:50 MQ H<sub>2</sub>O : reagent HCl) -- just enough to cover the bottom of the jars; 1-3 ml for vials

4-cap jar and place on hotplate at 80-100°C for at least 1 hr (best = overnight)

5-swirl and collect acid droplets at bottom

6-discard acid (ultimately into ‘HCl waste’ carboy) and rinse jar and cap twice with MQ H<sub>2</sub>O

- 7-add ~7 M HNO<sub>3</sub> (50:50 MQ H<sub>2</sub>O : reagent HNO<sub>3</sub>) -- just enough to cover the bottom of the jars; 1-3 ml for vials
- 8-cap jar and place on hotplate at 80-100°C for at least 1 hr (best if left overnight)
- 9-swirl and collect acid droplets from cap and walls
- 10-discard acid (ultimately into 'HNO<sub>3</sub> waste' carboy) and rinse jar twice with MQ H<sub>2</sub>O
- 11-if you are not immediately using the jars, store them in Ziploc bag or plastic bin

#### cleaning 15 ml polypropylene centrifuge tubes

- 1-add 1-2 ml 1x QD 1 M HNO<sub>3</sub>
- 2-cap and agitate
- 3-collect acid in a waste cup and discard into HNO<sub>3</sub> carboy
- 4-repeat with MQ H<sub>2</sub>O, and shake out all droplets
- 5-if a large number are cleaned in advance, store them in a Ziploc bag with an appropriate label

HF-HNO<sub>3</sub> silicate rock sample digestion This procedure is designed for silicate rocks of <100 mg size. Larger samples become difficult to avoid voluminous precipitates in the final product. Peridotites and similar high-Mg materials are ordinarily troublesome at the >50 mg volume level, as are organic-rich materials. Both of these are best dealt with using HClO<sub>4</sub> (no details are presented here for that procedure). **The end product of the digestion must be a completely clear solution.** Any residual solid (aside from trace phases like spinel) will harbor Li and induce isotopic fractionation.

Although the analytical procedure consumes ~X0 ng Li, it is always easiest in the long run to digest enough material for several separate measurements, unless it is a precious or particularly difficult (low abundance) sample. Hence, for N-MORB, 25 mg of sample is more than adequate.

When working with concentrated mineral acids be certain to use gloves and eye protection and wash your hands after handling vials (even when capped) containing HF. A set of <100 mg-size samples can usually be made to come to clear solutions within 3 days of beginning digestion, given regular supervision. Although the procedure is listed in these terms, samples frequently take longer to digest, particularly when not actively monitored.

#### **day 1:**

Before weighing samples, wipe the balance area with moistened Kimwipe. Place a sheet of Al foil to the right of the balance where samples and vials will be placed. Your notebook should not be on the balance table (adjacent to the sink or on a stool are convenient alternatives). Before using balance, delicately brush off pan and check that balance is properly leveled. Use one blast from the Zero-Stat gun to reduce static with vials. Vials can also be wrapped in Al foil to reduce static electricity effects.

Use a clean spatula to transfer sample powders into 15 ml Teflon screw-cap vials. Do not allow sample powder grains to disperse all over the inside of the vial, particularly in the threads. Grains in threads will carve grooves in the soft Teflon. Rinse spatula with Kimwipe and MQ H<sub>2</sub>O between samples and wrap with plastic film when finished. Unless you are spiking samples, a weight precise to single milligrams is all you need. Record all weights. Label vials on sides and top. Two separate labels on opposing sides of a vial are recommended, as ink can be accidentally wiped off during handling.

When dealing with dropper and squirt bottles, be careful not to touch tips to anything that is not verifiably clean, or to have material (solid or liquid) splash onto the tip. Think ahead about the sequence of events so as to avoid such incidents. Lay down a sheet of plastic film in the area where you are working to facilitate clean-up. Air flow in the laminar flow cabinets is left-to-right. It is best to work with samples on the downwind (right) side of the cabinet. Note: temperatures indicated are not strict, just general guides.

- 1-transfer samples for digestion to laminar flow cabinet #4, and set up the rectangular hot plate, keeping the controls on the upwind (left) end
- 2-add ~0.5 ml 1x QD conc. HNO<sub>3</sub> from the dropper bottle; this should be enough to completely cover and engulf sample powder
- 3-swirl sample so as to completely homogenize in the HNO<sub>3</sub> and remove sample dust from vial walls

- 4-while gently swirling the sample, add ~ 2 ml conc. HF from squirt bottle -- samples may have an instantaneous vigorous reaction as SiF<sub>4</sub> vapor is produced, so angle the vial away from you and never stick the bottle tip into the vial (be careful: this bottle squirts slightly to the right, rather than straight ahead)
- 5-if a squirt bottle tip comes in contact with sample or other material, set it aside and see Paul
- 6-continue to swirl, uncapped, for ~1 min
- 7-replace cap of vial and screw on very loosely (just enough to keep it on)
- 8-plate on hot plate at 60°C (just warm) for 1-2 hr
- 9-swirl vial again for ~1 min, focusing on breaking up any clumps of material; large clumps may be disaggregated with ~10 minutes in the ultrasonic bath with cap on tight
- 10-cap vial tightly and place on hot plate at 80°C overnight (24 hours is better, over weekend is ideal); periodically examine vial and swirl contents

**day 2:**

- 11-collect droplets from walls and cap by swirling vial, uncap and dry down at 90°C
- 12-when sample is dry, add 10-20 drops 1x QD conc. HNO<sub>3</sub> and swirl to break up sample cake; again, use ultrasonic bath (capped vials only) if large clumps fail to break up manually
- 13-dry completely at 90°C, repeat steps 12-13
- 14-when sample is dry, add 10-20 drops 1x QD conc. HCl and swirl to break up sample cake
- 15-over the course of ~3 hr, allow the sample to dry at 75°C to ~50% original volume; every 20-30 minutes add a few drops of conc. HCl and swirl vial – most samples at this point have traces of solid whitish material: the original silicate is gone and the solid here is most likely alkali earth-fluoride, which is most easily disrupted with HCl
- 16-when the liquid volume gets low (<0.5 ml) and if there are abundant trace solids, add another 10-20 drops 1x QD conc. HCl, cap tightly and place on hot plate at 80°C overnight (if this solution is completely clear, you can go on to the day 3 procedures; you can also choose to leave the sample in the current state for 24+ hours)

**day 3:**

- 17-uncap and repeat progressive HCl drying, steps 14-15
  - 18-when solids are gone, add ~1 ml MQ H<sub>2</sub>O – swirl and look very closely for solids: if none exist, take the sample to complete dryness; if there are solids, repeat the progressive HCl drying, or consider adding HCl and leaving capped for a few hours (as in step 16)
- (**note:** steps 19-20a are for **12 ml column chemistry**, 19-20b are for **three column chemistry**)
- 19 a-for samples with no traces of solids remaining, when the sample has just achieved dryness (looking somewhat resinous--do not dry excessively to a brown crust) add ~1 ml 1x QD 1.0 M HNO<sub>3</sub> – watch closely as sample dissolves: if any solid exists, add several drops of 1x QD conc. HCl and go back to step 15
  - 19 b-for samples with no traces of solids remaining, when the sample has just achieved dryness (looking somewhat resinous--do not dry excessively to a brown crust) add 1 ml 1x QD 4.0 M HCl (dropper bottle in laminar flow cabinet #4) – watch closely as sample dissolves: if any solid exists, add several drops of 1x QD conc. HCl and go back to step 15
  - 20 a-for completely clear solutions in 1.0 M HNO<sub>3</sub> (examine with sufficient backlighting to reveal even faint solids) transfer sample to the appropriate 15 ml centrifuge tube to a volume of 4 ml 1x QD 1.0 M HNO<sub>3</sub> and set aside for chemistry (**note:** some samples show tiny bits, say <0.01 ml, of insoluble material in 1.0 M HNO<sub>3</sub>, which disappears after sitting for some length of time... from ~1 hr to more than 12 hr; also note: samples at this stage can be left for weeks without concern of precipitation from solution)
  - 20 b-at this stage completely clear solutions in 4.0 M HCl are ready for the first column
  - 21-save the pipette tip for when the sample is added to its column

## \* \* 12 ml Column Chemistry \* \*

equilibrating columns This chemistry should take one whole day, with samples drying overnight. Column cleaning, unless begun immediately after samples come off the columns, takes about one day. Before beginning chemistry, always wipe the area with moistened Kimwipe (including columns stand, etc.). Lay a sheet or sheets of plastic film down in the working part of the area. Remember that space is limited, so do not spread items over more than the workspace and perhaps beside the sink (and there only when chemistry is ongoing).

If the resin bed has been allowed to dry out, it must be fully disaggregated using MQ H<sub>2</sub>O and the dedicated syringe-probe. After this, resin must be prepared for accepting samples by being treated with an nitric acid + methanol mixture. The equilibration mixture is 0.1 M HNO<sub>3</sub> in 80% methanol (abbreviated 0.1/80). The elution mixture is 1.0 M HNO<sub>3</sub> in 80% methanol (abbreviated 1.0/80). Consider that acid + organic waste should be separated from entirely-acid waste. There is a bottle specifically for nitric + methanol waste in the “organic waste” area.

Handle columns gently! When removed from the stand, be sure both column and gloved hand are dry to avoid them slipping from your grasp.

- 1-remove reservoir from columns; use small PMP beakers or blue centrifuge tube caps as temporary covers
- 2-if resin in columns has dried add ~10 ml MQ H<sub>2</sub>O
- 3-disaggregate bubbles in dry portion of resin using the syringe-probe dedicated to Li chemistry
- 4-allow resin to settle and H<sub>2</sub>O to pass through, ensuring that all bubbles have been eliminated
- 5-empty waste cup into sink
- 6-add 10 ml of 0.1/80 to each column and allow to pass
- 7-add another 10 ml of 0.1/80 to each column;
- 8-after resin settles, draw up most of the 0.1/80 into the syringe-probe and slide the probe into the resin – as you move the probe around in the resin, inject small amounts of 0.1/80 throughout the resin; watch as small bubbles are destroyed – manipulate the probe to ensure these flow up and out of the resin
- 9-examine resin in each column (especially the lower half) for bubbles – if bubbles persist, allow resin to settle, add enough 0.1/80 to fill the quartz glass portion of the column, and repeat syringe-probe step 8
- 10-after resin has settled and solution level is lower, add the remaining 4 ml of 0.1/80 to each column (each column should have ~24 ml of this solution applied to it before sample loading)
- 11-before storing the syringe-probe, rinse any acid from it by drawing up several ml of MQ H<sub>2</sub>O and expelling

readying samples Before beginning chemistry, calculate necessary volume of 1.0/80 to prepare (this reagent is made up fresh for each set of chemistry). The mixture should be allowed to return to room temperature before use (due to heat of mixing upon combination of methanol and polar solvents). For the calculation, consider that each column to be used will consume 140 ml 1.0/80 and that due to volume change on mixing, ~5% more than the minimum needed should be made. For a set of 8 columns, the following recipe can be used: *915 ml methanol + 191 ml 1x QD 6.0 M HNO<sub>3</sub> + 38 ml MQ H<sub>2</sub>O*. The 1.0/80 is mixed in the dedicated Teflon bottle, which is rinsed out after use with MQ H<sub>2</sub>O. If you choose to use concentrated HNO<sub>3</sub>, do not directly combine 100% methanol with concentrated HNO<sub>3</sub> (this is a potentially explosive mixture) – dilute the acid beforehand with H<sub>2</sub>O.

There is a 250 ml Teflon bottle and 60 ml screw-top vessel for the 0.1/80, which may not need to be mixed up freshly each time. Pipette from the screw-top container.

- 1-to each sample (in 15 ml centrifuge tube with 4 ml 1 M HNO<sub>3</sub>), add 3 ml TD methanol, to a final total volume of 7 ml – the solution is thus ~0.6 M HNO<sub>3</sub> in ~40% methanol
- 2-cap and agitate
- 3-centrifuge for ~10 minutes at maximum speed

Examine each solution before proceeding to columns. If visible material has formed, sample should be aborted (and taken back through digestion steps, beginning with #13). Insoluble material from the digestion (e.g., spinel, rutile, graphite) can be disregarded due to insignificant Li abundances, but **do not load them onto the resin**.

**Note:** If samples are to be taken through secondary chemistry (column #3 of the **three column chemistry**), the same 15 ml Teflon sample vials as used in the digestion should be cleaned and used for elution from the

secondary column. Thus, begin cleaning these immediately, as described above, so that vials will be ready the forthcoming morning.

loading samples Once the equilibration 0.1/80 solution has passed, samples can be loaded, keeping the same waste cup beneath the column. Remove the reservoir from each column. In adding the sample, stick the pipette tip down into the stem of the column, below the yellow adapter piece. Drop slowly to begin with so as not to disturb the resin bed. After 2-3 ml have been added, the remainder can be added more rapidly. If there is remnant solid material at the bottom of the centrifuge tube, do not add this to the column. Do not allow the sample solution to make contact with the adapter (where the glass column ends). This is to avoid possible contamination of subsequent samples.

Once the sample has passed into the resin, carefully drop in 1 ml 0.1/80. This is to scavenge any residual sample Li from the column walls. Add it slowly around the circumference of the stem, so as to hit everywhere along the walls that sample touched. Make sure to use a pipette tip dedicated to the purpose, and that is discarded if it touches any column walls.

cleaning sample centrifuge tubes After loading the sample, the centrifuge tube can be re-used. The pipette tip used to load the sample can be similarly cleaned and re-used.

- 1-add 1-2 ml 1x QD 1 M HNO<sub>3</sub> from squirt bottle to tube, cap and agitate
- 2-collect acid in a waste cup
- 3-add another 1-2 ml 1 M HNO<sub>3</sub>, draw this into the sample's pipette tip and expel into waste cup
- 4-discard acid waste into HNO<sub>3</sub> carboy
- 5-add 2-3 ml MQ H<sub>2</sub>O, cap and agitate
- 6-shake out all droplets
- 7-add another 2-3 ml MQ H<sub>2</sub>O, draw this into the sample's pipette tip and expel into waste cup
- 8-set tube and tip aside until sample is through chemistry and ready to be transferred

elution When the 1 ml rinse has passed through the resin, the column elution can begin. Several elements have lower affinity for the resin than Li, and are removed with an initial aliquot of 1.0/80, which is discarded. After this, Li is collected in clean Teflon jars. You may notice some small bubbles in the bottom several cm of the column during elution—this is an unavoidable consequence of the mixed acid + organic medium. As the Li elution progresses, this bubbles will dissipate.

To avoid disturbing the resin bed, pipette in the first 3-4 ml of solution, then it will be safe to carefully pour from the graduated cylinder. The yellow reservoirs must be attached tightly or they will leak. A tight seal is best achieved by a slight screwing motion when the reservoir is pressed into place.

- 1-add and discard 30 ml 1.0/80
- 2-in sequence, cover each Teflon jar with Parafilm so that the tip of the column just enters the jar along one edge, to minimize splashing (i.e., do not have columns dripping into the center of the jar)
- 3-place the appropriate 180 ml Teflon jar (labeled with sample or column #) beneath each column
- 4-collect Li with 110 ml 1.0/80 (this elution takes on the order of 5 hours to complete)

drying samples Drying down to small droplet on a hot plate at 80°C (in laminar flow cabinet #4) takes slightly more than 12 hours. Drying under a heat lamp takes on the order of 5 hours if positioned ~10 cm from a non-'dim' bulb. You should be present to watch as the last sample droplet comes to dryness. Avoid baking, as is easily the case if jars are left unattended at 100°C, as the residual organic component of this material may not be readily soluble. At this point most samples should go directly to secondary columns. If they are to be analyzed without secondary chemistry, calculate the volume of 0.33 M HNO<sub>3</sub> (= 2%) needed to dilute sample to 100 ppb Li and transfer into the appropriate 15 ml centrifuge tube. Always pipette from the vial of acid, not the bottle.

cleaning 12 ml columns As soon as a set of chemistry has been finished, column cleaning must be begun. The cleaning takes some time, so starting it right away will allow the next set of samples to be put through as soon as possible. Do not leave columns in  $\text{HNO}_3 > 1 \text{ M}$  overnight. Long life of the resin can be assured if it is not exposed to strong, concentrated oxidants for prolonged periods of time. If you need to leave and the columns contain  $> 1 \text{ M HNO}_3$ , wait until this passes and add  $\sim 15 \text{ ml MQ H}_2\text{O}$ .

1-add 50 ml MQ  $\text{H}_2\text{O}$  and discard waste

2-add  $\sim 10 \text{ ml 1x QD } \sim 7 \text{ M HNO}_3$  and wait about 5 minutes while the resin in suspension settles

3-when resin has settled, add 50 ml 1x QD  $\sim 7 \text{ M HNO}_3$

4-with the 1x QD 1.0 M  $\text{HNO}_3$  squirt bottle, rinse the tips of each column into waste cups

5-discard accumulated waste into  $\text{HNO}_3$  carboy (this ignores trace methanol in this solution)

6-with MQ  $\text{H}_2\text{O}$  squirt bottle, rinse the tips of each column into waste cups

7-add  $\sim 200 \text{ ml MQ H}_2\text{O}$  with graduated cylinder and squirt additional MQ  $\text{H}_2\text{O}$  over the remaining exposed internal surfaces of the reservoirs

miscellaneous after-chemistry chores

-After use, rinse all graduated cylinders with MQ  $\text{H}_2\text{O}$ , shake dry, cover with plastic film, return them to laminar flow cabinet #4.

-Discard unused 1.0/80 into the waste bottle and rinse the dedicated 1.0/80 bottle twice with MQ  $\text{H}_2\text{O}$ .

-Remove plastic film and wipe down countertops and laminar flow cabinet with moistened Kimwipes.

## **\*\* Three-Column Chemistry \*\***

overview These steps are intended to operate separately from the 12 ml methanol chemistry, although the third column of this procedure may be a useful 'clean-up' column for samples that have passed through the 12 ml columns but still have intolerable Na/Li. Each column is loaded with Bio-Rad AG 50w-x12 (200-400 mesh) resin. The clean stock of this resin is stored in MQ H<sub>2</sub>O. As always, all samples should be in 100% clear solutions and solids must never be loaded onto the columns. Centrifuge samples that are questionable and add solutions with care.

As with all chemical procedures, wipe down counter area with moist Kimwipe and lay down plastic film in the work area before beginning.

The first two columns are prefabricated from polypropylene (commonly called Bio-Rad columns). Resin should be filled to the 1 ml division. It is easy to disturb the resin bed with these columns, so be careful to add the first 1-2 ml of solution very gently. The third column is made from quartz glass and has a different internal diameter than the first two columns. The marks on the stems of these columns indicate the proper level for 1 ml of resin. The narrow stem of this column makes it possible to create an air bubble between the resin bed and the column bowl, effectively sealing off solution from the column. The bubble can be eliminated using the micropipette and a gel-loading pipette tip.

Dry resin must be thoroughly disaggregated before chemistry. Add 5-10 ml MQ H<sub>2</sub>O and wait for suspended resin to settle. Disaggregate dry resin using the dedicated syringe-probe (a small version of the one used with the 12 ml columns is kept on the shelf in the REE workstation for this task). If columns are to be used straight away, allow resin to settle and draw off excess H<sub>2</sub>O with syringe or pipette. Leave syringe-probe tool rinsed and dry after use.

first column This column step uses moderately dilute HCl. A clean 7 ml Teflon vial should be in place to **capture all effluent** from these columns, even that which comes off when the sample is loaded.

- 1-equilibrate columns with 1 ml 4.0 M HCl
- 2-dissolve samples in 1 ml of 4.0 M HCl and load drop by drop onto resin (remember to collect this solution)
- 3-collect an additional 3.5 ml 2.5 M HCl; set this to dry on hot plate or under heat lamp
- 5-begin column cleaning immediately so columns are ready for next user, collecting all HCl waste: add ~1 ml 1x QD ~6 M HCl drop by drop, and allow this to pass (acid above resin will commonly take on yellow color as Fe from resin diffuses back into solution)
- 6-add another ~1 ml ~6 M HCl drop by drop, and allow to pass
- 7-add ~10 ml ~6 M HCl, drop by drop at first, then more rapidly after solution volume protects resin from disturbance; during this stage rinse column tip with a few ml of ~6 M HCl (from squirt bottle)
- 8-when all HCl has passed, discard waste in HCl waste carboy
- 9-add ~10 ml MQ H<sub>2</sub>O (essentially a full reservoir)
- 10-rinse column tip with a few ml of H<sub>2</sub>O
- 11-after sample is dried, it is ready for the third column

second column This column step uses dilute HCl. A clean 22 ml Teflon vial can be used to collect the eluant from these columns.

- 1-equilibrate columns with 1 ml MQ H<sub>2</sub>O
- 2-dissolve samples in 2 ml of 0.15 M HCl and load drop by drop onto resin
- 3-add and discard 4 ml 0.15 M HCl
- 4-collect 20 ml 0.15 M HCl -- vials will be fairly full at the end, so be careful that the column tip is not placed far into the vial during elution; set this to dry on hot plate or under heat lamp
- 5-begin column cleaning immediately so columns are ready for next user, collecting all HCl waste: add ~1 ml 1x QD ~6 M HCl drop by drop, and allow this to pass (acid above resin will commonly take on yellow color as Fe from resin diffuses back into solution)
- 6-add another ~1 ml ~6 M HCl drop by drop, and allow to pass
- 7-add ~10 ml ~6 M HCl, drop by drop at first, then more rapidly after solution volume protects resin from disturbance; during this stage rinse column tip with a few ml of ~6 M HCl (from squirt bottle)
- 8-when all HCl has passed, discard waste in HCl waste carboy

- 9-add ~10 ml MQ H<sub>2</sub>O (essentially a full reservoir)
- 10-rinse column tip with a few ml of H<sub>2</sub>O
- 11-after sample is dried, it is ready for the third column

third column This chemistry employs a mixture of dilute HCl in ethanol. A clean 15 ml Teflon vial can be used to collect the eluant from these columns. A flow rate of 0.13 ml/min was measured in the initial set of columns.

- 1-equilibrate columns with 1 ml MQ H<sub>2</sub>O
- 2-dissolve sample in 1 ml 0.15 M HCl for loading; avoid sample solution entering the bowl portion of the column--load sample in 2-3 aliquots
- 3-add and discard 2 ml 0.5/30
- 4-collect Li in 10 ml 0.5/30 -- vials will be fairly full at the end, so be careful that the column tip is not placed far into the vial during elution (this elution takes on the order of 4 hours to complete); set this to dry on hot plate or under heat lamp
- 5-begin column cleaning immediately so columns are ready for next user: add 10 ml 1x QD ~6 M HCl, drop by drop at first then more rapidly after solution volume protects resin from disturbance; during this stage rinse column tip with a few ml of ~6 M HCl (from squirt bottle)
- 6-when all HCl has passed, discard waste in HCl waste carboy (this ignores trace ethanol in this solution)
- 7-add ~15 ml MQ H<sub>2</sub>O (essentially a full bowl)
- 8-rinse column tip with a few ml of H<sub>2</sub>O
- 9-once sample has dried, calculate the volume of 0.33 M HNO<sub>3</sub> (= 2%) needed to dilute sample to 100 ppb Li and transfer into the appropriate 15 ml centrifuge tube

miscellaneous after-chemistry chores

- After use, rinse all graduated cylinders with MQ H<sub>2</sub>O, shake dry, cover with plastic film, return them to laminar flow cabinet #4.
- Discard unused 1.0/80 into the waste bottle and rinse the dedicated 1.0/80 bottle twice with MQ H<sub>2</sub>O.
- Remove plastic film and wipe down countertops and laminar flow cabinet with moistened Kimwipes.

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