



## Cleaning Procedures for Laboratory Material used for Chemical Preparation/Column Separation in Lead Isotope Analysis

Abbreviations:

- MQ water: Ultrapure water („Milli-Q“ water)
- \*\*\* = triple-distilled

### 1. Cleaning reagent vessels/vials

- ☐ Rinse three times with MQ water
- ☐ Fill with 6%  $\text{HNO}_3$ , make sure that no air is trapped in beakers/vials, let it sit for at least 1 night
- ☐ Empty  $\text{HNO}_3$ , fill with MQ water, let it sit for at least 1 night
- ☐ Store until needed

### 2. Cleaning Teflon (“Savillex”) beakers

#### 2.1 Pre-cleaning

- ☐ Place beakers in large beaker and fill it with a mixture of lab detergent (e.g., DECONEX) and deionised water, let it sit for at least 1 night
- ☐ Rinse three times with deionised water
- ☐ Fill the large beaker with Aqua Regia (4/8 deionised water, 3/8  $\text{HCl}$  p.a., 1/8  $\text{HNO}_3$  p.a.), let it sit for at least 24 h on a hot plate at approx. 70–80 °C
- ☐ Rinse three times with deionised water
- ☐ Rinse once with MQ water
- ☐ Fill the large beaker with 6N  $\text{HCl}$ , let it sit for at least 24 h on a hot plate at approx. 70–80 °C
- ☐ Rinse three times with MQ water
- ☐ Take beakers out of large beaker and let them dry

#### 2.2 Bleaching

- ☐ Fill 1 ml 6M  $\text{HNO}_3$ \*\*\* into the individual beakers, screw tightly, leave for at least one week on a hot plate
- ☐ Rinse three times with MQ water
- ☐ Fill with MQ water and add a few drops of  $\text{HBr}$  (to bind the lead), leave it for at least one night or until needed
- ☐ Dry beakers at least one day before needed (they should be cooled down to room temperature for weighing)
- ☐ Store until needed

### 3. Cleaning columns

- ☐ Allow columns filled with old resin to dry
- ☐ Empty used resin (tap or rinse with water using a pipette repeatedly)
- ☐ Rinse with MQ water
- ☐ Place in sealable plastic container with a mixture of lab detergent (e.g., DECONEX) and deionised water (make sure no air is trapped in the columns) and shake closed container
- ☐ Rinse three times with MQ water
- ☐ Put columns in large beaker filled with Aqua Regia (4/8 deionised water, 3/8  $\text{HCl}$  p.a., 1/8  $\text{HNO}_3$  p.a.), let it sit for at least 24 h on a hot plate at approx. 70–80 °C
- ☐ Rinse three times with MQ water
- ☐ Fill the large beaker with 6N  $\text{HCl}$ , let it sit for at least 24 h on a hot plate at approx. 70–80 °C
- ☐ Rinse three times with MQ water
- ☐ Take columns out of beaker and store them 5%  $\text{HCl}$ \*\*\* for at least 1 week or until needed
- ☐ Rinse with MQ water before use





#### 4. Cleaning new resin (dry product)

- ☐ Pour some spoons of resin (e.g., DOWEX AG1x8 or other in use) into a 1 L bottle
- ☐ Fill up to approx. 500 ml with MQ water and 10 ml 6M HCl\*\*\*
- ☐ Shake several times
- ☐ Decant
- ☐ Repeat multiple times
- ☐ Fill up again with approx. 500 ml of MQ water and 10 ml 6M HCl\*\*\*
- ☐ Store like this until needed

These procedures were adopted from the former isotope laboratory of Friedrich Begemann, Max-Planck-Institut für Chemie, Dept. Isotopenkosmologie, Mainz, Germany.

