## Silica gel emitter preparation

Procedure modified from Gerstenburger and Haase, 1997, Chem. Geol.

#### Materials

0.2 g colloidal silicic acid (Merck)

19.6 g 0.035M H3PO4 (diluted from concentrated orthophosphoric acid)

#### Method

Using an acid-washed 250µl pipet tip and a clean pipettor, draw approximately 200 µl of colloidal silicic acid from the very top of the undisturbed 30 ml bottle of stock solution, and weigh into a tared, empty, clean 7 ml Savillex beaker.

Dilute the colloidal silicic acid with 0.035M H3PO4 (make sure the molarity of the dilute phosphoric acid is accurate by titration) in the weight ratio, 0.2 g silicic acid : 19.6 g 0.035M H3PO4. To do so, weigh the appropriate amount of 0.035M H3PO4 into a 30ml spaghetti tube-tipped dropper bottle, and place it in an ultrasonic bath. With your clean pipet tip, pipet the weighed colloidal silicic acid from your beaker into the bottle dropwise, allowing the silicic to disperse under sonication.

The resulting dilute silica gel emitter should yield loading blanks less than 200 fg Pb, and ion currents of greater than 10,000 cps per pg (measured on 205Pb spiked loading blanks).

It is recommended to sonicate the bottle of silica gel solution regularly to maintain the dispersed silica in solution.

If the silica gel solution yields anomalously high blanks, then it can be cleaned by treatment with coarse anion exchange resin in a dilute HBr medium as follows:

1. To the ~20 ml of silica gel solution mixed above, add 1.11 mL (1.65 g) of conc. (9M HBr) to yield a 0.5M HBr solution.
2. Starting with dry AG1-X8 20-50 mesh resin, immerse a batch in MQH2O in a small bottle (e.g. 250 mL), shake and let the coarsest resin settle before decanting the fines. Repeat the filling, shaking and decanting of the fines until you have a well-sized coarse resin fraction.
3. Add enough clean 6M HCl to the bottle to cover the resin, let equilibrate for an hour, the decant the 6M HCl. Repeat this process several times, alternating MQH2O and 6M HCl. Finish this procedure with several rinses of MQH2O prior to storing the resin in MQH2O.
4. Add approximately 2 mL of wet, sized, clean resin to the silica gel mixture in 0.5M HBr and recap. Shake this bottle occasionally over the course of a day and let sit overnight, or place in an automatic shaker for 24 hours.
5. The next day, decant the silica-gel mixture into a new dropper bottle, being careful not to transfer any resin to the new bottle. The cleaned silica gel should have loading blanks reduced by at least 50% compared to the starting composition.