Directions for running low level Ortho-Phosphate samples on the Alpkem

All glassware and bottles used should be acid washed before beginning.

Check that the instrument is configured to run PO_4. The line from the sampler should be connected to the pump tube labeled "PO_4 sample" and lines labeled "PO_4 to spec" and "PO_4 from debubbler" should be connected to the channel 3 debubbler. If not, see the section: "Switching the Alpkem from NH_4/NO_3 to PO_4".

Empty the large DI water bottle and fill with 1.2 M HCl from the acid bath. Let it sit for at least 15 minutes. Carefully pour the acid back into the acid bath, rinse 6 times with DI, and fill with DI.

Dissolve 1.8 grams ascorbic acid in about 70 mls DI water in a 100 ml volumetric flask. Dilute to 100 mls with DI water.

Check sampler wash waste reservoir under bench to right of instrument. Empty into sink if more than half full.

Turn on main power switch and make sure PO_4 heater is on.

Open Winflow 4.0 software. If software is already open, exit and reopen. This causes the sample probe to go into the wash reservoir

Lock down the pump platens and pull the engaging levers straight up for all the pump tubes labeled PO_4 plus "to sampler wash" and "from sampler wash".

Press the "run stop" button on the pump, the display should read –50.0.

Connect the sampler wash line to the port labeled sampler wash on the large DI water bottle. Connect the PO_4 color reagent line to the port labeled NO_3 carrier on the large DI water bottle (be sure to connect to the ports as listed: the sampler wash will not flow properly if connected to the port labeled NO_3 carrier).

Connect the "PO_4 Dowfax" line to the dowfax bottle.

Let the system pump for 15 minutes and check for leaks.

Prepare the color reagent by adding the following stock solutions to the color reagent bottle IN ORDER and mixing after each addition:

150 mls 5N sulfuric acid
15 mls antimony potassium tartrate solution
45 mls ammonium molybdate solution
90 mls ascorbic acid solution

If there are no leaks, move the PO_4 color reagent line to the color reagent bottle.

Let the system pump 15 minutes before running samples.

Setting up a sample run

Press the sample table button on the main tool bar in Winflow4.

Type in "cup #", "name" and "type" for each sample and standard to be analyzed. Refer to the typical sample table on the page 5 and the notes below.

Cup Numbers:

Positions 1-20 are the large tubes at the back of the sampler and are used for standards, checks and baselines (washes). The instrument can sample from these cups up to 16 times each.

There are 3 racks for the 2 ml sample cups. The one on the left (closest to the sampler wash cup) holds cup numbers 101-190. The middle rack holds cup numbers 201-290 and the rack on the right holds number 301-390. These cups can be sampled up to 4 times each.

Sample Name

Sample names can be anything that fits in the field. However, the standards used in the calibration curve (anything labeled "C" in the "type" column) must be named exactly the same as they are named in the calibrant table in the method file.

To view the calibrant table, click the "edit method" button on the main toolbar, click file, open, and select LOPO4.mth. The calibrant table is the last page of the method file.

Type

SYNC-This is always the first cup in a run, it is a high standard (usually top standard) which will yield a large peak to let the software know the sample peaks have started.

C- indicates a calibration standard to be included in the calibration curve. Do not use this for internal standards or checks unless you want them included in the calibration curve.

RB- indicates a baseline (wash). The instrument will use these peaks to correct for baseline drift so they should always be a 0 ppm standard.

U- unknown. Should be used for all samples, checks, and internal standards.

After the calibration curve, the basic pattern should be 10 samples, 1 standard, 10 samples, 1 baseline. Repeat to a maximum of about 80 samples. Larger sets should be broken into multiple runs. Sample runs should always end with a baseline.

Other columns in the sample table

R- Number of replicate samples to be taken from the cup. Should be set to 1.

Dil- dilution factor of the sample, usually 1.

Wt- weight of the sample, usually 1.

After the sample table is prepared, be sure to save it.

Starting a sample run

Click the collect data button on the main toolbar. The software will ask for an operator name and ID, type in your initials for both. The software will then ask for a sample table name and a method name. The method name is LOPO4.mth. Select the correct file names and press OK. The software will ask for a filename for the results with the default being the same as the sample table with an .rst extension. Select or type in a file name and press OK. The software will not allow you to overwrite or append and existing file.

The data collection window will appear on the screen. Press the "play" button to start the data collection. At this point, the software will monitor the baselines for 60 minutes and then start sampling.

Debubble the flow cell by pinching and releasing the outflow tubing. Repeat until no bubbles appear.

Pour standards and samples into the cups and place in the sampler. Get at least 20 cups poured before starting the sampler.

Check baselines for stability and drift, should be less than 500 micro-absorbance units.

If there are any peaks or jumps in the baseline traces, Press the "stop" button. The software will ask if you want to stop, press yes. Press the "rewind" button and then the "play" button.

Press the "fast forward" button to begin sampling.

Instrument shutdown

Move the "PO_4 color reagent" line to the "NO_3 carrier" port on the large DI water bottle. Let the system pump for about 10 minutes.

Disconnect all the lines and let the system pump air for about 15 minutes or until all the liquid is out of the instrument.

Press the "run stop" button to stop the pump and turn off the main power switch to the instrument.

Push the engaging levers on all the pump platens to horizontal and disconnect one end of each platen from the pump.

Pour the unused color reagent into the PO_4 waste container.

Empty the PO_4 waste into the container in the acid cabinet in A258.

Empty and discard the sample cups and tubes. Rinse the sampler racks with DI water and set on the bench to dry.

Refrigerate the standards if the instrument will be used the next day or freeze them if it will be longer.

Wipe down the instrument and benches with a moist sponge.

Switching the Alpkem from NH_4/NO_3 to PO_4

Make sure the NH_4 waste container is empty. The waste goes in the bottle on the floor by the sink.

Disconnect the "NH_4 waste from spec" line from the outflow tube of the channel 3 (PO_4/NH_4) detector and connect the "PO_4 waste from spec" line in its place.

Disconnect the "NH_4 to spec" line from the bottom connection on the debubbler on the channel 3 detector and replace it with the "PO_4 to spec" line.

Disconnect the "NH_4 from debubbler" line from the top connection on the debubbler and replace it with the "PO_4 from debubbler" line.

Turn the NH_4 heater off and the PO_4 heater on.

Disconnect the sampler line from the "T" where the "NH_4 sample" line and the "NO_3 sample pull thru" line meet and connect it to the pump tube labeled "PO_4 sample".