Directions for running NO_2 analyses on the Alpkem (4/28/15 DER)

Check that the instrument is configured for NH_4/NO_3: The line from the sampler should be connected to the "T" where the NH_4 sample line and the NO_3 pull through line meet. The debubbler on the channel 3 detector (NH_4/ PO_4) should be connected to one line labeled "NH_4 from debubbler" and another line labeled "NH_4 to spec". If not, see the section: Switching the Alpkem from PO_4 to NH_4/NO_3.

Remove NO_3 color reagent bottle from the fridge and place it in a warm water bath to warm up to room temperature.

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

Turn on compressed air at the red handled ball on the bottom of the tank regulator.

Turn on main power switch.

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 NO_3 plus "to sampler wash" and "from sampler wash".

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

Empty, rinse and refill the large DI water bottle for the sampler wash and NO3 carrier..

Connect the sampler wash and NO_3 carrier lines to the large DI wash bottle (be sure to connect to the ports as labeled: the sampler wash will not flow properly if connected to the port labeled NO_3 carrier).

Connect the NO_3 lines: color reagent and buffer, to the NO_3 rinse bottle (DI water).

Let pump for 10 minutes and check for leaks.

Move the NO_3 buffer line to the buffer bottle.

Move the NO3 color reagent line to the color reagent bottle.

Make sure the line from the sampler is completely clear of all other lines and the reagent bottles so the sample probe can move freely.

Let the system pump at least 15 minutes before beginning a sample run.

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 7 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 6 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 should only be sampled one time 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 nh4no3.mth. The calibrant table is the last page of the method file.

The format for naming calibration standards is: X where X is the concentration of NO_2. No leading zeros on values less than 1 and no decimal points except for fractional concentrations.

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 nitrite.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 both flow cells 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.

The first NO_2 peak will appear about 1 minute after the first sample is drawn.

Instrument shutdown

Move the NO_3 lines to the NO_3 rinse bottle. Let pump 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 NO_3 waste into the container on the floor next to the instrument.

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

Return the NO_3 color reagent bottle to the fridge.

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.

Turn off the compressed air.

Switching the Alpkem from PO_4 to NH_4/NO_3

Make sure the PO_4 waste container is empty. The waste goes in the bottle on the bottom shelf of the acid cabinet in room A258.

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

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

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

Turn the PO_4 heater off and the NH_4 heater on.

Disconnect the sampler line from pump tube labeled "PO_4 sample" and connect it to the "T" where the "NH_4 sample" line and the "NO3 sample pull thru" line meet.