January 17, 2017

OFFICE OF WATER QUALITY Water-Quality Information Note 2017.03

SUBJECT: Alert and preliminary guidance for addressing nitrogen contamination of Pall Versapor GWV High Capacity capsule filters.

PURPOSE:

The purpose of this Water-Quality Information (WaQI) Note is to alert all USGS field personnel who filter water-quality samples with a Pall Versapor GWV High Capacity capsule filter (NRSS, One Stop item number Q398FLD) of the likelihood for nitrogen contamination. The National Water Quality Laboratory (NWQL) has identified a positive bias in measurements of dissolved Kjeldahl nitrogen (DKN) and total filtered nitrogen attributable to contamination of water samples by the Pall capsule filters that are stored after rinsing for any period of time. Storing pre-conditioned filters for as little as one hour prior to sampling may create a positive bias for DKN and total filtered nitrogen. Chilling the pre-conditioned filters is not adequate to prevent this bias from occurring. No bias was observed when sample filtration was completed immediately following conditioning.

BACKGROUND:

The current version of the National Field Manual (NFM) states in section 3.3.4.A, that capsule filters are to be pre-rinsed with Type I deionized water (DIW) to prepare them for filtering water-quality samples and that the pre-conditioned filters can be stored chilled for up to 1 day (USGS, various dates). However, in response to recent information that this practice may result in contamination of samples for nitrogen analyses, the NWQL has completed preliminary testing of the effects of storing the wet filters. Ten capsule filters were pre-rinsed with the prescribed 2 liters of DIW and stored in sealed plastic bags either at room temperature (5 filters) or at 4 +/- 2 degrees Celsius (5 filters). Storage times were initial (t=0), 1 hour, 4 hours, 24 hours, 4 days, and 7 days. After the designated storage time, capsule filters were rinsed with 20 mL of water and then used to collect samples for nutrient analyses. After sample collection, the capsule filters were resealed in plastic bags and stored until the next sample-collection time. Laboratory-prepared Nanopure water was used for both rinses as well as the sample water.

Samples were collected from all ten capsule filters at t=0 (no storage) and analyzed for the following labcodes: 1985, 2331, 2754, 3116, 3117, 3118, and 3157. All results for t=0 were below the corresponding detection limits.

Preliminary results for DKN (LC1985, pcode 00623) and total filtered nitrogen by alkaline persulfate (LC2754, pcode 62854) show a trend of increasing DKN and total filtered nitrogen contamination in as little as one hour after conditioning (fig. 1). Results for other analytes including nitrate/nitrite (LC3157, pcode 00631), nitrite (LC3117, pcode 00613), ammonia (LC3116, pcode 00608), total phosphorus (LC2331, pcode 00666), and orthophosphate (LC3118, pcode 00671) were all below the associated method detection limits. These preliminary results suggest that the primary contaminant under these testing conditions is an organic nitrogen compound that is leaching from the filter membrane or binder in the capsule filter. The NWQL is conducting additional testing to determine the effects of acid rinsing the filters. NWQL and OWQ will be evaluating the results and will issue a report and an OWQ Technical Memorandum as soon as testing is complete. The National Field Manual will be updated and a publication is planned.

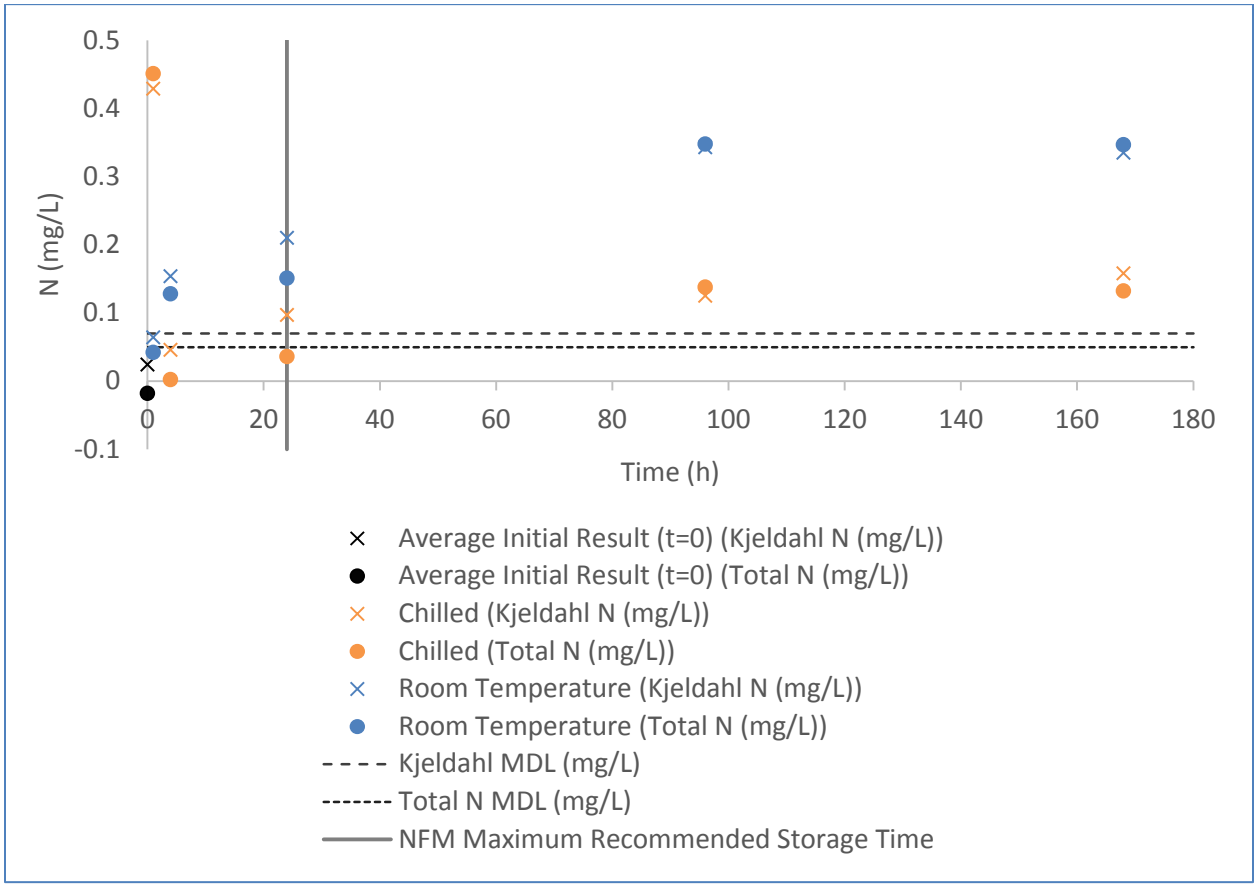

Figure 1. Total nitrogen and total Kjeldahl nitrogen (mg/L as N) from pre-rinsed filters with varying storage times and temperatures. Orange markers are from filters stored at 4ºC prior to sampling, blue markers represent filters stored at room temperature, and black markers are at t=0 (no storage). Times for collecting samples from the capsule filters were Initial (t=0), 1 hour, 4 hours, 24 hours, 4 days, and 7 days.

INTERIM GUIDELINES:

Effective immediately, Pall Versapor GWV High Capacity capsule filters must be used ***immediately after*** conditioning with the required 2 liters of IBW (inorganic-free blank water from One Stop) or DIW (Type I deionized water, 18 MΩ water that has been quality assured). It is no longer recommended that the Pall Versapor GWV High Capacity capsule filters be stored for any length of time after rinsing, even if they are chilled. This modified protocol is required for all filtered/dissolved analytes that use the Pall Versapor GWV High Capacity capsule filter until further testing is complete.

Please continue to collect quality-control samples with your environmental data to quality assure your results. No capsule filters other than the Pall Versapor GWV High Capacity have been tested at NFSS at this point, so please use caution when going to sources outside of NFSS.

Contact Cherie Miller (cvmiller@usgs.gov) if you have any questions.

WaQI Notes are archived on the Office of Water Quality web site,
http://water.usgs.gov/usgs/owq/WaQI/index.html

Signed,

The Office of Water Quality

This memorandum supersedes cleaning and filtration procedures in the National Field Manual, chapter A3 section 3.3.4.A and chapter A5 section 5.2.1.A, which allowed pre-rinsed capsule filters to be stored chilled for up to 24 hours.

Distribution: All WMA Employees

REFERENCES:

U.S. Geological Survey, variously dated, National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chaps. A1-A10, available online at http://pubs.water.usgs.gov/twri9A.