### **Data Integrity Workgroup Lab and Analysis Meeting Minutes**

The meeting was recorded for note taking purposes.

# 1:30PM-3:30PM Meeting Materials Link

#### **ACTIONS**

- ✓ Durga Ghosh (USGS) will connect with Pamela Higgins (PA DEP) to discuss bacterial monitoring and analysis.
- ✓ Anyone who is interested in switching from Lachat and wants to test out other instruments may reach out to Chesapeake Biological Laboratory (CBL) and visit them to test out their instruments. Contact: Kim Blodnikar (blodnika@umces.edu)
- ✓ Durga Ghosh and Jerry Frank (CBL) will discuss how Jerry can best get Durga the blind audit data.
- ✓ Amy Goldfischer (CRC) will set up a Teams site for the group to communicate over in between meetings.

Participants: Amy Goldfischer (CRC), Ian McMullen (DNREC), Jerry Frank (CBL), Suzanne Doughten (ODU), Cindy Johnson (VADEQ), Heather Wright (ODU), Jaclyn Mantell (CBL), Jay Armstrong (DCLS), Lara Phillips (MDH), Chengyuan Cao (MDH), Keri Maull (DNREC), Durga Ghosh (USGS), Laura Lockard (DNREC), Lexis Carter (ODU), Pamela Higgins (PA DEP), Liz Chudoba (Alliance for the Chesapeake Bay), Tracee Cain (DNREC), Najma Khokhar (MDE), Scott Schroeder (FIALabs), Kim Blodnikar (CBL), Jake Kilczewski (CBL), Gary Sunday (Sun Analytical), Alexandra Fries (UMCES), Sergio Huerta (DNREC)

### 1:30 PM Nutrient instrument analyzers

**Suzanne Doughten** 

• Discussion of what instruments laboratories are currently using, and if they are planning on changing/ordering a new system.

Suzanne Doughten commented that most of the people she knew of were using a Lachat flow injection analyzer and Lachat is being discontinued. She was wondering who is still using it and who is replacing it. They're still using it but looking into replacing it and haven't found a replacement that they're thrilled with.

Kim Blodnikar (CBL) said they never used a Lachat. They are using the discrete analyzers for their higher-level concentrations with the enzyme method for nitrate and all of their other inorganics like ammonium, orthophosphates, and nitrate. When it comes to low level cadmium nitrate, they were using the Segmented Flow Auto Analyzer IIs (AA2) still up until just last month when they switched to an AA500 and they love it. It is also segmented flow like the AA2. It is a slightly different technology from Lachat but they could not be more pleased with the support they received from Seal Analytical, the company it's from, and the weeklong training they provided. It's been a quick changeover to use it. Granted, Kim had already been using segmented flow so a lot of

that was interchangeable although the software is new to Kim. It's also hitting below their previous detection limit. Kim thinks it will be in-between the initial Method Detection Limit (MDL) and what they used to have as a continual MDL. Jay Armstrong has the same instrument.

Suzanne asked if it made the standards for Kim? Kim said that she makes all her own standards. The standards she made for the AA2 are completely interchangeable. She makes her own reagent. She's only using it for cadmium nitrate. However, it is an option to have the instrument make columns for you.

Jay Armstrong commented that Division of Consolidated Laboratory Services (DCLS) also purchased one of these instruments last year. The flow injection has been nice because of how well it handles the variable salinities, and this instrument does the same thing. The capillary flow cell and fluidics in this instrument run faster than the old segmented flows. Once they get into running samples on this, the sample progression is just as quick as the Lachat, about a minute to 90 seconds, once the samples start hitting the detector. DCLS is looking at setting it up for all the parameters. They're working on the initial data right now. Detection limits are similar. The software has been a little different; he's used Skalar software and HACH software and this is different. It's just a matter of figuring out how different manufacturers phrase different things. Thus far DCLS has been pleased with what they've seen. That said, Jay added, they don't handle the range of salinities that they handle at Old Dominion University (ODU) and CBL. DCLS has a narrower range of what is submitted to them.

Suzanne asked, does it have the ability to make the standards and do the autodilution like the Lachat did? Jay replied that yes it does, but he is not using it to make standards because he doesn't know that syringe set up is going to make sub ppb level standards as accurately as they can make them with volumetric pipettes. They're used to making their own standards. Even with the Lachat, if the analyst makes the standards the instrument runs through it faster by an hour or so, because it takes so long for the Lachat to make the standards. That's a separate set up program to set up the instruments for a certain set of predilutions that is run as a dilution and run file that's used to set up runs. There would have to be a separate dilution and run file to do a set of standards because doing a post dilution requires a separate dilution and run file . Jay said that DCLS does have that set up for doing post dilution with this. They haven't made use of that because they're just getting into collecting what they need, but Jay did test it out and was pleased with what he saw. He said that for anything that will dilute over range he did feel comfortable that the dilutor will form correctly. When running the dilution run or something like that it is important to put some kind of check standard in to make sure the diluter is running faster.

Suzanne asked if it is a lot quicker than the Skalar? Jay responded when he first started doing this type of work in 1997 they were still outfitted with Technicons AA2 and one Skalar. The AA500 is similar to the Technicon AA2 except it electronically sets peak heights and gains rather than manually. They still have 4 Skalars there and their instrumentation is very durable. Of every sampling, it takes about 2 minutes and 20

seconds. With the Seal, the wash time is not as much so it cuts down on the amount of time between samples.

Kim said that the Technicon AA2 ran about 40 samples an hour and she is running 55 samples an hour. However, it's possible to customize it in the software to go faster or slower depending on rotation and wash times. Jay said if he's running all 4 channels at once it does 40 samples an hour. Jay added that an AA500 is set up with an automatic shutdown which the Lachats don't have.

Suzanne asked if it could automatically shut down with a cadmium column on it? Kim said that yes, it could, and it is possible to customize the shutdown procedure. Part of that customized and automated procedure is to turn off the valve. There's nothing to physically turn to shut off for the cadmium column. It has a light on the top to indicate when it's on. It's possible to have multiple different shut down procedures depending on what you're doing that day. Jay said it was a similar premise to the pinch valve to set the manifold on the Skalars for the nitrate channel. He said that it's not a column in the sense of what they were used to on the Lachat and Skalars. It's a cadmium coil that's covered in a plastic. It is then hooked into the system. It's activated with copper sulfate just like you would activate cadmium granules. Initially he was concerned about that because they had experience with that on another instrument and the columns didn't hold up well, but on this instrument it has been very resilient. The other thing about that is you have to have a nitrogen source available. Instead of having a separate air pump, you're introducing the air through a set of pinch valves connected to a pump tube. You have a bag that you fill with nitrogen, which is pumped through the column. Then, the only way you introduce air into the column is if you do a dry sampling, and Jay found it's pretty resilient to that. Having the nitrogen injected through it helps with its life between regeneration.

Kim said she makes her own column.

Jay clarified that they fill the nitrogen bag from a nitrogen tank. He said in the manual they explain how to pack one of the columns into a tube.

Heather said when they went from Skalar to Lachat it was a toss-up between Seal and Lachat. They ended up going with Lachat because they thought the maintenance would be easier. Although they don't have to replace the glass tubing pieces the cost of the Lachat maintenance has been more than anticipated. Heather asked when it dilutes after the run can you add your extra QC that's needed to close out?

Jay said that when they come to do the install, make sure they set up your dilution files for you and they can program into that dilution file what that table will be. That table will throw in blanks and continuing calibration verifications in your dilution run if you have it programmed the right way with tags in the run. When it does a dilution run it's different from the Skalar and the Lachat.

Heather said it sounded like it would be easier for her to manually dilute whatever she needed to. Jay said it depends. If it's going to run overnight than having the diluter is a

viable option. However, if you can be there during the day when it's running you can dilute and add to your table faster than the instrument will do it for you because what will happen is the instrument will finish a run and generate a table to run anything that you program in as a shoulder peak. For instance if you have a sample above a certain threshold and it's labelled as a shoulder peak, it will rerun it. Then the run stops, and then it makes up its new table for what all the dilutions will be. It does that automatically. The diluter is making the dilution run rather slow, but it's automated. It will make that and run its primer (similar to a tracer in the Skalar) which will tell you where your first peak is and the timing of your other peaks based on that. It'll run your primer and the drift and then it goes into dilution. That entire process will be automated, and then it will do an automatic shutdown. As long as you program everything, it will be all automatic.

Jay asked is there a reason Kim went with the granular cadmium? Kim said she's been making her own columns for 20 years so it wasn't any different than making one for the AA2. It was an easier transition for her to not worry about the bag of nitrogen and enabled her to get it up and running as soon as possible. Jay asked how to make the column – is it with a piece of Tygon tubing and you put a glass wool on the end? Kim said yes, and it goes through a whole cleaning process with acetone and an HCL process, and a copper sulfate process rinsing off the extra. Then you pack it into 0.110 Tygon tubing that's filled with water. Then you add the glass wool at the end, and spend a day or two conditioning it. Jay said once you make one, do you regenerate in the tube? Jay found they regenerate them in the column and it works great. Kim said she hasn't done a regeneration on hers but does things in its lifetime to preserve it such as replacing the glass wool, and repacking it and cutting off the excess. Her columns last 6-9 months depending on what's she's running. Kim said they do some of their nitrate analysis through enzyme and it depends on the time of year how many samples they run; currently a couple hundred per weeks. Jay said the old procedures beginning with Technicons, you would unpack that column, do all the regeneration, regenerate the cadmium in a beaker with copper, rinse it out and put it back into the column it originally came out of. It proved more safe and efficient to strip the cadmium while it's in the column and regenerate it while it's in the column using the syringe. If you're losing efficiency you might be able to pick up your efficiency that way. Kim said in their ammonium chloride they do put copper sulfate in there – 3 drops per 500 milliliters and keep that in there as part of the life preservation of the column. Jay said his lab doesn't do that.

Suzanne asked if they have any kind of expiration dates, because there's still life in her column but have to put the new one on before the expiration date and she feels like it's a waste. Kim said that she doesn't have an expiration date; she goes by reduction efficiency and instrument numbers. What she notices first is her reference material starts dropping, expected concentration vs actual concentration starts dropping. Jay said to Suzanne she could pack her own into the Lachat tube. She said she's thought about it; she used to pack the Skalar ones and it wasn't a big deal. Jay said he thought it was an open by date. Suzanne said she called them and confirmed that it is an open by date (not an expiration date).

Jay described the nitrogen bag – he said imagine a small IV bag with a gas valve. That's all it is and you hook it up to a piece of Tygon tubing connected to a nitrogen cylinder, put gas in the bag, close the valve, take the bag to the instrument, and hook it up to Tygon tubing connected to the pump that pumps it through the column. Then all your segment bubbles are nitrogen. When they go through the column it doesn't hurt the column. That's another difference between Kim's setup and Jay's setup. Since Kim is using a granular column that she's packing then she'll have a de-bubbler before it goes into the column. With the setup Jay's lab ordered and will be using, there is no de-bubbling going into the column, so it can't be air going into the column which is why they have the nitrogen. The nitrogen setup is so the channel moves a little faster. All the nitrogen bubbles go through the column. Some things to consider when talking with the sales representative: unlike with the Skalar work, the flow cells were de-bubbled on Skalars and the bore of the flow cell was much bigger. For Seals, and it will remain to be seen how much it takes to make sure the timing remains right, these flow cells are not de-bubbled. The way this works is all of the air bubbles or nitrogen bubbles pass through a flow cell and through a timing mechanism, those are timed out of the signal. The signal is averaged for when it's reading the actual liquid in the cell and that's based on the timing from the pump. One of the reasons these are faster is they're bubble through technology instead of de-bubbled technology. Supposedly that's supposed to help with refractive index issues as well.

Keri Maull (DNREC) said she wanted to give input from their lab. They do still have a Lachat and run TP, TN on it and they will fully transition to a new instrument for that. They also have two older Seal models which they love. If they get something new it will probably a new Seal. They also have two Astoria Pacifica continuous flows that they use for nitrate nitrite and for OP. Tracee Cain is their inorganic chemist and can answer questions. They're on the same page about the Seal. They are great with customer and tech support. Jay said they haven't set up a service contract yet. How do you like your service contract? Keri said it's good. They have a PM contract – they just service the instruments. They don't have a disaster contract or anything. The technicians that come out are awesome, they answer all your questions.

Jay said if you have a component failure or something like that with the Lachat contract you'll either get a full component repair or replacement. It looked to Jay when he was reviewing the Seal contract you get 10-20% off and one PM and one emergency visit a year. Keri said that in the 10-20 years she's been there they haven't had any disasters. They have older models and are looking for something new to go with. They have issues with Astorias although they do send new components when there is an issue. They haven't had a lot of major breakdowns with the Seal. Jerry commented in the chat that they have multiple Das(Discreet Analyzer) and would love to get a new one in the next year or so, but their favorite instrument (thermo Aquakem 250) was discontinued several years ago.

Jay said that older models of Seal used filament type halogen bulbs and this AA500 uses led lights sources tuned to the wavelength of whatever you're testing. Gone are the special wavelength filters you have to replace. Gone are yearly bulb replacements. For all the great things flow injection did it ate reagents like nothing else.

Jerry Frank asked if anyone in this group is using a discrete analyzer other than CBL? CBL was the only lab doing so. Jerry asked, who was using a DI carrier or saline? Jay said DI. Kim said DI carrier, and they have a broad range of salinities and are not having issues with it. Jay said that not having to debubble the flow cell and looking at a capillary size flow cell cuts down on the refractive issues. He would recommend you call them up and send them your most challenging samples.

Kim said to Suzanne that if anyone in her group wants to come up to CBL and wants to play around with the instrument and demo, feel free to email Kim. Lara Phillips asked, does anyone know what the price comparison is for discrete vs alternatives? Heather said she can't speak to discrete, but her lab's quotes from Seal lab was around 60k but that included a smaller sampler; 5 modules, and a diluter would have been an extra 9k. Quote from Seal was 53k in December 2020 for 3 channel AA500, and 60k for a 4 channel. Jay said he thought his quote was around \$70k for a 4 channel with a diluter and a 300 sampler.

Lara said that her lab has been using Lachats but is looking into alternatives. They just don't know where the funding is going to come from for the new instrument. Heather said she didn't know if anyone else has had success but she was bugging the sales rep for OI analytics but after 3-4 months gave up. If the sales person doesn't respond, customer service isn't going to be good. Pam said that her lab currently has the Lachat, Skalar and OI Analytical. OI Analytical has the worst customer service. They had some big problems with software and autosamplers glitching that they couldn't fix. They have been looking into the Seals. They do have some ion chromatographs (ICs) but they're used as a backup for lower volume. Jerry said they had an OI carbon analyzer back in the day. This morning's meeting was a good example of why these types of meeting are needed as well and he felt like the time was well spent. The group was able to discuss this analyzer issue at length.

#### 2:00 PM Open discussion

Cindy asked how often should this lab meeting happen? Jerry suggested planning to have it twice a year, but go to quarterly if there are other issues the group wants to discuss that come up.

Durga asked Jerry about blind audits – she asked if 2020 is the last set he uploaded to his website? Jerry said when CBL was audited for their certification this spring and their plan was to get all of their updated SOPs and the most recent blind audit materials, etc, uploaded at the same time. If FY21 isn't up there, that's an oversight. Jerry said he'd check and get that up there. Jerry said he can have a discussion with Durga on how he can get the data to her in a way that's most useful. If he can just provide the data and some of the simpler analysis that they do, Durga can take the data for a deeper dive if needed.

Durga asked if there was anyone in the group who does bacterial analysis and if she could reach out to them about the methods they use and other criteria. Pam Higgins said they do at PA DEP and can answer questions. Durga said they're struggling with certification and questions regarding that. Does PA require any kind of certification? Not just for bacterial work but in general lab work?

Pam responded that for environmental lab work the state requires certification for e coli, total coliforms, fecal coliform, Heterotrophic Plate Counts (HPCs). They're the only lab in PA that does giardia and crypto testing. That requires EPA certification and is a one-year analyst training, and you have to renew your certification every year with online testing. Pam said they have recently been offering qPCR for molecular source tracking, and ELISAs for cyanotoxin testing. Their biological services have exploded in the recent years. They are accredited by the state for the EPA methods for cyanobacteria testing. Some are in house because EPA doesn't have any. They end up with some inhouse accreditations as well and they do that as the primacy lab for PA. They are very familiar with accreditation.

Durga said she'll reach out to Pam and explained there are a few citizen monitoring groups she's working with MDE on their IR, and there's always questions about the usefulness of the data collected by citizen monitoring groups. She'd like to see how they compare to programs in VA and MD. Jay said that their lab is doing similar work and Durga can contact him as well.

The final topic discussed was supply availability. Jerry said it's horrible. The supplies he can get have quintupled in price. Jay said they were having a hard time getting potassium sulfate. Jerry said for a long time they had a shortage with gloves from the PPE shortage. Then potassium hydroxide. Sodium hydroxide liquid they are still waiting on, they keep bumping the delivery date. He found an alternative source through another manufacturer. Finding supplies is getting a lot harder and taking more time. Cindy said she'll find out if the bottle issue has cleared up in another month; the last time, it took 4 months to get 250 mL bottles. Jay asked if she wants people to hold on to some bottles? Cindy said perhaps although that's not their preferred method; they had some high hits on the blanks. In order to re-use bottles they have to do a cleaning study on their blanks to see if they're adequate.

Keri said that their Fisher representative Rebecca finds them stuff they think is gone. They're under state contract with places so they can't shop around. Pam said it's the same in PA, although it's VWR, not Fisher. They're not as good as finding alternatives. They do have a couple small colleges in their area and as an adjunct professor she knows some of them. Sometimes they reach out to the colleges to get potassium sulfate or a common salt to tide them over when they're really desperate.

Jerry said they go to VWR, Thomas, and SPC in Canada. Just recently they learned of Ibis Scientific and they have had what they needed and it's been cheaper than VWR. They supply the Teflon filters used for chlorophyll. It might be worth checking them out. Cindy said most of their vendors have to be Small, Women or Minority Owned Businesses which makes it more difficult to find. Jerry said that their discrete analyzers

are Thermos instruments. He thought they were the only source of the cuvettes. Each cuvette has 12 cells. Those are things that have quintupled in price over the last 3 years. However, he found a company in Italy that makes them more cheaply. He ran them through tests and they seem to be analytically equivalent. But the university and state of MD discouraged buying from Italy. Cindy shared that EPA now has a mandate for American built supplies.

The meeting concluded with a brief discussion of communication between meetings and decision to use a Teams site to ask each other questions between meetings.

## 3:30 PM Adjourn