Top Tips for Conducting DMR-QA Analyses

Presenter: Tom Widera

Inorganics Product Line Manager
ERA – A Waters Company

DMR-QA 34 Dates & Deadlines

- Opens Monday, Friday, March 21
- Closes Midnight, Friday, July 11
- PT providers send graded reports by August 1
- Permittees send report to DMR-QA coordinator by August 25
- Corrective Action reports due by October 10

Outline

- DMRQA Study and Dates
- Acceptance Limits
- Tips for Running TSS
- Tips for Running pH
- Questions

DMR-QA 34 Dates & Deadlines

- If running WP, report results by close of WP study.
  - WP study results are returned within 2 business days of the close of the study. (ERA is the only provider to have results in 2 days; due to the enormous size of the DMR-QA study, DMR-QA results will be available in less than 21 days
- If you choose to use a WP study for DMR-QA, the study must close between January 1 and July 11.

	Opens	Closes	Results
WP 228	Jan 13	Feb 27	Mar 4
WP 229	Feb 17	Apr 3	Apr 8
WP 230	Mar 10	Apr 24	Apr 29
WP 231	Apr 14	May 29	Jun 3
WP 232	May 12	Jun 26	Jul 1
DMR-QA	Mar 21	Jul 11	Jul 25

DMR-QA 34 Acceptance Limits

- Based upon EPA regression equations
- Find regression equations at http://www.nelac-institute.org/fopt.php
- 3 standard deviations/99% CI around an expected recovery
- Concentration dependent
- Expected recovery = assigned value * a + b
- Expected standard deviation = assigned value * c + d

DMR-QA 34 Acceptance Limits

EPA Regression Equation for Total Suspended Solids (TSS)

a - 0.9728; b - (-0.6338); c - 0.0300; d – 1.5793

at 23 mg/L limits = 64.8 – 124%

at 100 mg/L limits = 82.9 – 110%

DMR-QA 34 Acceptance Limits

Example Calculation MINIMUM TSS Value

Made at 23 mg/L

Expected Recovery = Value * a + b

23 * 0.9728 +(-0.6338) = 21.74

1 Expected Standard Deviation (SD) = Value * c + d

23 * 0.0300 +1.5793 = 2.269

3SD = 3 * 2.269 = 6.81

Limits = Expected Recovery ± 3SD = 21.74 – 6.81 = 14.9

DMR-QA 34 Acceptance Limits

Example Calculation MAXIMUM TSS Value

Made at 100 mg/L

Expected Recovery = Value * a + b

100 * 0.9728 +(-0.6338) = 96.65

1 Expected Standard Deviation (SD) = Value * c + d

100 * 0.0300 +1.5793 = 4.579

3SD = 3 * 4.579 = 13.7

Limits = Expected Recovery ± 3SD = 96.65 + 13.7 = 110

Total Suspended Solids

1. Use enough sample
2. Rinse apparatus thoroughly
3. Dry thoroughly
4. Minimize environmental controls
5. Run filter blank

Total Suspended Solids

- Report proper units (mg/L)
- Use no less than a 4 place balance.
- When using gravimetric tests, ensure that you can accurately and appropriately read the amount of residue left in the pan.
- Environmental controls play a big role in the accuracy of TSS measurements. The more mass of residue that is on the balance, the less these environmental controls come into play.
  - Balance Uncertainty
  - Moisture gain or loss

Total Suspended Solids

- Dry your pans and filter paper completely prior to aliquoting samples. As per the method, Use 1.5 µm filter paper (Whatman 934-AH, or equivalent) placed into a light weight aluminum dish (or gooch crucible). The dish you use should be as light as possible to minimize tare weight.
- TSS powder will stick to everything. Make sure you rinse all glassware that comes into contact with TSS thoroughly with DI water. Mix the sample extremely well as the TSS is heavier than water and will tend to sink. Rinse the graduated cylinder and vacuum filtration apparatus at least three times with DI water to ensure no TSS residue sticks.
- Gooch crucibles vs. aluminum pans
- Moisture is an issue with TSS. Run a blank filter to determine that you are not gaining or losing moisture in the test.

Tips for Running TSS

Allow sufficient time for the sample to cool. As a general rule allow the crucibles to cool for at least an hour.

- Ceramic crucibles take longer to cool than aluminum pans.
- Although the crucibles may feel cool to the touch, if not completely cooled readings will be affected.
- Crucibles will cool at different rates.

Tips for Running TSS

- Ensure that the sample is completely dry. Dry the sample using your normal procedure. Once dry weight is measured, place the sample back in the oven for at least 30 minutes and reweigh to ensure that a constant weight is achieved. Cool for equal amounts of time for each part of the process.
- Use a desiccator to ensure that you are not introducing moisture when cooling.
- Wear gloves. Your hands contain oils and if transferred to the weighing dishes can falsely add weight to the dishes.
- Calibrate your balances every day you use them to ensure accuracy. It is recommended to calibrate the balances with weights that will bracket the weights you are measuring.

DMR-QA 34 Acceptance Limits

EPA Regression Equation for pH

Hard Limits at ± 0.2 s.u.

at 5.00 s.u. limits = 96.0 - 104%

at 10.0 s.u. limits = 98.0 – 102%

pH – Tips and Tricks

- Although the simplest of tests, pH has extremely tight limits. Limits are less than 5%. Ensuring that your equipment is running optimally is critical for success.
- Ensure that your values are temperature compensated. Automated temperature compensation (ATC) is necessary. It is critical to use your ATC probe and ensure that the ATC probe is calibrated. Refer to your owners manual for how to calibrate the ATC. A 1^{o} change in temperature can change your values by as much as 5%.
- The ATC can only accurately account for about a 5^{o} change in temperature. Make sure before you run your pH that you warm your buffers and samples to room temperature.

pH – Tips and Tricks

- Maintain proper operation of your probe. Clean once a month. Filling solution can supersaturate and film builds on the outside. Use either hot soapy water or 1% nitric acid for the outside. Clean the inside with DI.
- Check the operation weekly. A ten fold change in concentration will result in a 57 mV change. Run and record both the mV readings for the 4 and 7 buffer. The difference in readings should be 171 mV (allow a tolerance of ± 9 mV). If outside this range perform maintainance.
- The 10 buffer is very susceptible to CO_2 adsorption. Over short periods of time, the 10 will drift from air coming into the sample. This can be checked by using the same procedure above. Run and record the mV readings for the 10. The difference between the 7 and 10 should also be 171 mV.

pH Tips and Tricks – pH Check Example

- Place your probe into the pH 7 buffer. Select mV mode. The value should read 0 ± 30 mV. Place your probe in a pH 4 buffer. The mV reading should be 171 ± 9 mV difference from your 7.
- Example
  - pH 7 = 5.20 mV
  - pH 4 = 179.60 mv
  - Difference = 174.40 mV
- This shows the probe is working properly. You can then check the quality of your 10 buffer the same way. The 10 buffer should have a mV reading difference of - 171 ± 9 mV rom the 7 buffer. If it is outside of this range it is time to get a fresh pH 10 buffer.

pH – Tips and Tricks

- It is recommended to use the 10 buffer for only about 3 months after opening. A way around this is to use a cubitainer with a spigot on the bottom.
- Always use fresh buffers. Never pour the buffers back into the bottle after use as CO_2 adsorption and/or contamination can occur. pH buffers are relatively inexpensive.
- Calibrate your probe every day of use. The probes only drift slightly over time but may drift as much as 0.2-0.3 s.u. over a week. Daily calibration will prevent this drift.
- Calibrate with the smallest dynamic range. You need to bracket the pH of the sample but it is recommended to calibrate with only 2 buffers. A recommendation would be to screen the sample first to determine which 2 buffers to use.

pH – Tips and Tricks

- Make sure your readings stabilize. It should take no more than 30 seconds to obtain a stable reading. Any longer is an indication that the probe needs maintenance.
- Stirring vs. not stirring.
- Mix the sample well but do not agitate too much to prevent air bubbles from entering the sample.

Avoid Surprises

- Run analyses as soon as possible
- Allow for things to go wrong
- Establish a routine quality control process with the use of Certified Reference Materials

QUESTIONS

Contact ERA

800.372.0122

303.431.8454

6:00 am – 6:00 pm Mon-Thu (MT)

6:00 am – 5:00 pm Fri (MT)

info@eraqc.com

www.eraqc.com

www.facebook.com/eraqc

www.youtube.com/eraqc