

DMR-QA Technical Tips

Total Suspended Solids (TSS)

The purpose of measuring TSS is to make sure levels of suspended solids do not surmount to a point where the water can no longer support aquatic life. TSS analysis is one of the simplest tests to perform but sometimes the easiest things can be overlooked. Below are a few simple things to help ensure success with your TSS analysis.

- Make sure to filter a sample size that is large enough to accurately measure on the filter paper yet small enough to dry efficiently. The best range to reach this balance is between 10-200 mg of residue.
- Always use a 4-place analytical balance for best practice when weighing filters before and after use.
- Remember to use deionized water for filtering a blank sample.
- All pans and filter papers should be completely dried before use. It's advised to store them in desiccators after they have been dried and also before they are weighed.
- To ensure your filtered samples are completely dry, make sure to dry and weigh each sample at least twice.

Oil and Grease

Oil and Grease is a gravimetric analysis with some similarities to TSS. A few of the same tips and some unique steps apply to this analysis. As with TSS, it is important to use a 4-place analytical balance for every weighing. Unlike TSS, where you choose the sample size to filter, the entire oil and grease sample should always be used to ensure that all of the analyte is captured. You also need to rinse the bottle and cap three times with solvent to ensure that none of the sample remains attached to the container.

- Make sure that the pans/vessels are dried before aliquoting samples.
- Make sure you use the entire sample. Make sure you rinse both the bottle and cap three times with hexane and add the rinses to the separatory funnel.
- Cool pans/vessels in desiccators.
- Make sure you remove any water from the collection pans. Water contains dissolved solids and could cause the analysis to have a high bias.
- It is important to use a 4-place analytical balance.
- Dry and weigh each sample twice to make sure that all the moisture was removed in the first drying.

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Biochemical Oxygen Demand (BOD)

The BOD test determines the amount of dissolved oxygen used by microorganisms over a 5 day period. This is one of the most questioned tests among laboratories and so it's crucial that there be a high level of attention to detail for each step and take into consideration the following tips.

Control your contamination sources

- Residual chlorine found in tap water and organic material found in dirty glassware can affect your analysis. To avoid impacting your analysis be sure all glassware and equipment is meticulously cleaned and rinsed at least three times with deionized water.

Maintain a pH range of 6.5-7.5 for successful analysis

- BOD samples are always slightly acidic and the pH must be adjusted prior to analytical dilutions, otherwise you run the risk of a failing sample.
- Adjust the pH by adding ~0.2 molar NaOH, one drop at a time and checking with the pH meter that the pH criteria has been met.
- Be patient, it may take several minutes to adjust the pH.

Temperature

- Incubator temperature remains at 20+/-1°C – it is highly recommended you use an independent thermometer.
- Higher temperatures will increase the aerobic activity and thus increase the oxygen depletion.
- Lower temperature will decrease aerobic activity and thus decrease aerobic activity.

Total Residual Chlorine

The analysis of residual chlorine has the potential to be easily corrupted due to contamination from chlorine present in tap water and the volatility of the analyte. However, by taking a few precautionary steps, you can be assured of a successful analysis.

- Your glassware should be cleaned and rinsed at least three times with deionized water before your analysis.
- Always analyze a blank to ensure no contamination from your reagent water or glassware is present.
- Samples should be analyzed as soon as possible. Calibration standards and quality control (QC) samples should be diluted just before they are to be analyzed.
- Make sure you are using new, fresh reagents. For DPD reagent packets (powder pillows) make sure that the concentration range of the powder pillow is appropriate for the sample under test.
- If a 1mL pipette does not fit into a concentrate ampoule, use a clean, gas-tight syringe that is capable of delivering 1mL of concentrate instead.

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pH

Identifying the correct pH may seem as a simple procedure and analysis, but there are many factors involved that can affect the true measure of your pH. To ensure accuracy, always consider the slightest details that can help maintain the integrity of your pH measurement.

Quality Control

- Use a quality control sample to prevent usage of bad buffers or faulty probes. This can be done with a secondary source of pH to help verify your equipment and solutions are at a quality standard.

Temperature

- Analyzing samples for pH with varying temperatures can cause varying results.
- To control for variability and maintain consistency, run calibrations and check samples of pH at the same temperatures you run pH buffers.
- Most pH meters are equipped with ATC (Automatic Temperature Compensation) units to compensate up to about 5°C. However, if the temperature difference between the buffers and the samples is too great the ATC is rendered ineffective.

Calibration

- Be sure to calibrate your pH meter each time you perform analysis.
- Follow the manufacturer's instructions for calibrations.
- **Before you calibrate:** It is recommended to screen the pH sample to get an idea of what the pH will be. Depending on the screening results, calibrate the meter with the two buffers whose range is closer to the screening results.
- Always pour a small sample of your pH calibration buffer rather than sticking your probe in the buffer bottle

Use Fresh Buffers

- Through time, as buffers and samples become exposed to air during regular use, they become susceptible to carbon dioxide (CO₂) adsorption. CO₂ when adsorbed by a solution can become acidic and drop the pH of your buffer or samples.
- Make sure to follow the manufacturer's recommendations for storage.

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pH *(Continued)*

Cleaning your Electrode

- Clean your electrode at least once a month to remove impurities that may adulterate your results.
- Inside of your Electrode
 - Remove stopper and shake out solution
 - Rinse the inside of the probe with deionized water 2x
 - Rinse the inside with filling solution and then fill with fresh solution
- Outside of your Electrode
 - Place the probe in 10% nitric acid solution for 1 hour or
 - Place in a beaker filled with deionized water and sonicate for 15 minutes

Additional pH Tips

- Shake the sample and buffers well before aliquoting for pH analysis or calibration.
- Never return used buffer to the stock bottle.
- Allow ample time for the samples to equilibrate on the meter.