

**EPA DMR-QA STUDY PROGRAM
REQUIREMENTS AND LABORATORY PROCEDURES**

**TRAINING SEMINAR FOR WASTE WATER
MANAGERS AND LABORATORY ANALYSTS**

INDIANA 2011

NOTES

WHAT IS DMRQA

- DMRQA is an acronym which stands for Discharge Monitoring Report Quality Assurance.
- DMRQA is a Proficiency Testing (PT) program. This PT program is required for all discharge facilities which require a NPDES discharge permit.
- NPDES (National Pollutant Discharge Elimination System) permits are required for any facility which discharges pollutants from a point source into any waters of the United States. This includes most Waste Water Treatment Plants.
- The NPDES permits indicate the specific pollutants that must be monitored prior to discharging into the environment.
- Prior to discharging the pollutants contained on the NPDES permit, the effluent must be tested to ensure that the pollutants being discharged into the environment are below the maximum contaminant levels (MCLs) listed on the permit.
- To ensure that the testing is accurate the DMRQA program requires that the lab reporting NPDES data be certified to report this data.
- This means the lab needs to be able to prove that what it says is in the effluent is actually in the effluent.
- The DMRQA study is a series of PT samples which contain all the pollutants of interest. The values of the contaminants in the PT samples are unknown to the testing labs. The labs must analyze the samples using the same methods and procedures that are used to test the discharge effluent. The labs then report back their findings. The lab findings must reflect the actual concentrations of the pollutants in the PT samples within a given tolerance.
- DMRQA is conducted only once per year. The DMRQA study begins with the release of the 308a letter. PT samples are then ordered, analyzed and data are reported to the provider of the samples for scoring. The data and reports are then forwarded to the respective state or regional DMRQA coordinators.
- Non-compliance with the requirements of DMRQA can result in fines and/or revocation of the discharge permit. Most non-compliances can be rectified with some sort of corrective action.

NEW FOR 2011

There are very few changes for 2011.

- The response to the Address Verification Form must be delivered to the state DMRQA coordinator and not the EPA.
- State Certification Exemptions. There are several states that have applied for and received exemptions from the DMRQA program because the laboratories are required to complete a state certification program. The states are California, Nevada, North Carolina, Pennsylvania, South Carolina, West Virginia, and Wisconsin.
- In 2010, DMRQA added the requirement for analyzing Low Level Mercury at a concentration range of 20-100 ng/L (parts per trillion)
- In 2010, DMRQA added the requirement for analyzing Low Level Total Residual Chlorine at a concentration range of 75-250 µg/L (parts per billion)
- In 2009, DMRQA changed the reporting requirements so that all in-house and contract labs will report their test results directly to the PT provider. No one will forward their ungraded data to their state DMRQA coordinator. Permit holders will only send the final graded reports, EPA cover pages, and checklists to the state DMRQA coordinator.

DMRQA31 SCHEDULE

- March 14, 2011 – Response to the Address Verification Form is due
- March 14, 2011 – DMRQA31 begins
- July 1, 2011 – DMRQA31 study closes. Results are required to be reported to the PT providers by midnight in the time zone where the PT provider resides.
- July 22, 2011 – PT providers are required to supply graded PT reports to all laboratories.
- August 5, 2011 – Contract laboratories are required to forward one copy of their graded PT report to the permit holder.
- August 19, 2011 – Final reports are due to the state DMRQA Coordinator. The final report includes EPA Cover Pages, graded PT reports for all in-house and contract labs, and checklists for each lab supplying data.
- September 30, 2011 – Corrective Action reports are due for all “Not Acceptable” results.

THE 308A PACKET AND LETTER

- If you are required to participate in the DMRQA study, then usually in early spring (February or March) you will receive a packet from the EPA. (This is the 308A letter).
- The packet will contain all the information necessary for you to participate in the DMRQA study.
- The first page is your notice. **YOU MUST RESPOND** to this notice. By responding, you are letting your state coordinator know that you have received this notice. The response can be mailed or emailed to your state coordinator. You must send one response back for each permit you have.
- Next is your 308A letter. The 308A letter officially opens the DMRQA study. It details to you who must participate (you, if you received this letter), your responsibilities, the program tests, and where to get further information.
- Next is a checklist for your use. The checklist will give you required dates for each portion of the DMRQA process.
 - March 14, 2011 – DMRQA31 opening date. Address Verification Form response required.
 - July 1, 2011 – DMRQA31 Closing Date. Data needs to be reported to PT provider by midnight in the time zone where the PT provider resides.
 - July 22, 2011 – Graded Reports are due from the PT providers.
 - August 5, 2011 – In-house and Contract Labs need to forward a copy of the graded PT report to the permit holder.
 - August 19, 2011 – Permit Holder must forward one copy of the graded PT report, chemistry/microbiology and WET Checklist, EPA Cover pages to their state coordinator.
 - September 30, 2011 – all “Not Acceptable” results must be addressed and corrected and completed and signed corrective action report must be delivered to the state coordinator.
- Next is the DMRQA31 Fact Sheet.
- Next are the instructions for what you, as the permit holder, must do for DMRQA. Followed by what the contract labs must do.
- Next are the same instructions for the Whole Effluent Toxicity (WET) tests.
- Next is the list of the currently approved PT providers. These providers are approved by NIST/NVLAP to provide samples to you for the DMRQA study.
- Next is the list of state and regional coordinators for the DMRQA study.
- Next are the EPA cover page forms that need to be returned with the data reporting forms containing the analytical results from your lab, both in-house and contract.
- Finally, are the Chemistry and WETT Checklists to be sent to your coordinator with the graded PT results sent to you by your PT provider.

THE DMRQA PROCESS/ORDERING YOUR SAMPLES

- As soon as you receive the 308A letter from the EPA, initiate the process of ordering your samples.
- If you have a contract lab working for you, contact them to coordinate the sample ordering for your analytes. It is important that you and all of your contract labs are on the same page. Remember, it is your permit. Therefore, you are responsible for all phases of the process. Don't assume that your lab will take care of things for you. Make sure your contract lab knows what analytes they are reporting for you.
- Order your samples early. All samples will be shipped on the opening day of the study regardless of when they were ordered.
- When you receive the samples, check your package immediately. Make sure all of your samples are present, all paperwork is present, and that nothing has broken. Report any problems back to your PT provider immediately, so they can be rectified.
- If you or your contract labs have a requirement for Water Pollution (WP) certification as well as DMRQA, you can kill two birds with one stone. There are WP studies which will satisfy both requirements.
- The other benefit of using WP studies is that they close before the DMRQA study closes. Therefore, if corrective actions are required, they can be initiated even before DMRQA reports have been issued.

THE DMRQA PROCESS/ANALYZING SAMPLES

- CONDUCT YOUR ANALYSES EARLY.
- There are many problems that can occur during the analysis of your DMRQA samples. The earlier you conduct your analyses, the more time you will have if you run into problems.

THE DMRQA PROCESS/REPORTING YOUR DATA

- You are responsible for reporting all of your data to the PT provider. Your contract labs will be responsible for reporting their data to the PT provider on your behalf.
- ERAs online eDATA™ data entry system will simplify this process incredibly.
- With ERA, you can report your results via hardcopy data or on-line using ERAs eDATA™ on-line data entry system.
- For your lab results, fill out the data reporting sheets along with any necessary cover pages to the appropriate PT provider.

Hardcopy reporting of the final data package to your coordinator:

- Make sure you have all the data necessary for the analytes on your permit.
- You will need to submit the EPA cover forms along with your results to your PT provider by the close of the DMRQA study.
- You must submit one separate report for each permit you have. This includes submitting separate data reporting forms

- You will have two forms from the EPA that need to be included.
- **ERA will provide all of this information preprinted on the forms. If the information printed by ERA is correct, no manual entry is required. You only need to manually enter information on the EPA forms if there is an error.**
- The first page is the NPDES Permittee Data Report Form.
- Fill out your NPDES permit number and an extension if applicable.
- Fill out the Permittee Name. This is the official name that appears on your permit.
- If applicable, fill out the facility name.
- Fill out the facility address, including the city, state, and zip code. This is the address where your report will be sent.
- Fill in the name, title, fax number, email, and telephone number of the certifying official. The certifying official is the one who is responsible for the results. You, as the permittee, designate who this person is. An address is only needed when it is different from the facility address.
- The bottom of this form must be signed by the certifying official.
- The second EPA page is a list of all labs who reported data for your permit. You must include your in-house lab as well.
- You must list the USEPA lab code for each lab, including your in-house lab. Every lab has an EPA lab code.
- If you do not know your lab code, you can obtain this information from the EPA. It is in your DMRQA packet from the EPA.
- Indicate whether the lab did Chemistry, Microbiology, or WETT.
- Then indicate which classification the lab falls under.
- Then fill out the appropriate checklist for each lab that reported results for you. There is one checklist for Chemistry/Microbiology and one checklist for WET.
- **You must fill one checklist out for each lab that reported data for your permit.**
- For each checklist, indicate the analyte(s) that the lab reported for you. Then indicate whether the associated analyte received an Acceptable or Not Acceptable evaluation in the appropriate study.
- Include a copy of the graded report for those analytes and lab with the checklist.
- Repeat this process for all labs.
- Send this entire package to your coordinator.

THE DMRQA PROCESS/REPORTING YOUR DATA ON-LINE

- On-line data entry simplified the process tremendously. Plus you do not have to worry about whether your fax went through, or whether the mail got to the provider on time. On-line entry also reduces your costs as you are not worrying about express shipping late reports.
- ERAs on-line data entry system, eDATA™, simplifies the process even more.
- All data associated with your permit is entered under your permit number. Your contract lab will report their data directly to the PT provider.
- You enter the eDATA™ system under your ERA account number. Enter the results for the analytes run by your in-house lab. Your contract lab will report their results to the PT provider.

THE DMRQA PROCESS/CORRECTIVE ACTIONS

- If your test results yield a “Not Acceptable” result for any analyte on your permit, you will be required to perform a corrective action by the date listed in the EPA packet. DMRQA31 Corrective Actions are due September 30, 2011.
- The corrective action will consist of a few steps.
- Investigate your process, to try to determine why the results were “Not Acceptable”.
- Put measures in place to correct the problem.
- Obtain a Quality Control (QC) Sample, QuikResponse™ (QR) or enroll in a WP study. Remedial samples are not required for math or transcription errors.
- Write up your corrective actions in a report.
- Send this report along with your acceptable results package to Becky Ruark..
- If you are having trouble finding the cause of your problem, call ERAs technical staff. Our technical staff will spend as much time with you as necessary to help you fix your problem.

DMRQA 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

EPA REGRESSION EQUATIONS

O&G a - 0.9400; b - (-0.4116); c - 0.0545; d - 2.0789
at 20 mg/L limits = 44.4 – 140%
at 100 mg/L limits = 71 – 116%

BOD a - 0.6312; b - 0.1919; c - 0.1032; d - 0.167
at 15 mg/L limits = 30.0 – 98.7%
at 250 mg/L limits = 32.0 – 94.4%

CBOD a - 0.5423; b - 0.2956; c - 0.0996; d - 0.0697
at 15 mg/L limits = 24.9 – 87.3%
at 250 mg/L limits = 24.9 – 84.4%

NH3-N a - 0.9866; b - 0.0806; c - 0.0775; d - 0.0738
at 0.65 mg/L limits = 53.7 – 168%
at 19 mg/L limits = 74.7 – 124%

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%

TRC a - 0.9643; b - 0.0186; c - 0.0848; d - 0.0027
at 0.5 mg/L limits = 73.0 – 127%
at 3.0 mg/L limits = 71.3 – 123%

pH ± 0.2 s.u.

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

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

$$100 * 0.0300 + 1.5793 = 4.579$$

$$3SD = 3 * 4.579 = 13.7$$

$$\text{Limits} = \text{Expected Recovery} \pm 3SD = 96.65 + 13.7 = 110$$

HARDNESS/TSS ANALYSIS

- If possible, wear gloves. Your hands contain oils that if transferred to the weighing dishes can falsely add weight to the dishes.
- If available, it is highly recommended to calibrate your balances each day you use them. This will help ensure accuracy of the balances. It is recommended to calibrate the balances with weights that will bracket the weights you are measuring each day.
- Dry your pans and filter paper completely prior to aliquoting samples. Use 0.45 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. The heavier the dish weight, the more weight you will have to tare out when weighing residue. In order to ensure the accuracy required a 4-place (or analytical) balance is necessary.
- Dry the pans for at least one hour. Place the pans in a dessicator to cool to protect it from gaining moisture. Cool the pans for a minimum of 30 minutes (1 hour for gooch crucibles). Obtain a weight of the empty pan and filter paper.
- Filter the sample using a vacuum filtration apparatus. Make sure you rinse out the container used for the sample volume at least three times with deionized water. Place the rinses through the filtration apparatus. Then make sure you rinse down the sides of the vacuum filtration apparatus in case some of the residue is on the side.
- Make sure a blank sample is dried. Use deionized water for the blank. The blank sample is used to assess whether there is any moisture gain or loss during the analysis.
- Dry the sample in an oven at 105-120°C for at least two hours. Remove the pan from the oven and place in the dessicator to cool. Cool for a minimum of 30 minutes. (It is advisable to cool for the same amount of time as was cooled before the initial weighing).
- Place the sample back into the oven for an additional 30 minutes. Place the sample back into the dessicator to cool. Reweigh the sample. This is to ensure that all moisture was removed during the initial drying period.
- It is important when calculating the final result that the amount of weight gained or lost in the blank sample is compensated for. If the weight difference is negative add this weight to the sample. If the weight is positive, add this weigh to the sample.
- Divide the gram weight measured for the residue by the volume of sample used (in liters) then multiply by 1000 to obtain a result in mg/L.

TIPS AND TRICKS

- Run QC samples. These are samples that you know the certified value of and are provided with acceptance limits. For DMRQA they can be used as practice samples for you to determine how well your analysis is going. They are also great for use in your routine Quality Control program.
- Make sure you use enough sample so that the residue on the filter paper is enough to accurately measure.
- Choosing the amount of sample to be used is critical. A good estimate of the amount of residue can be assessed. The minimum concentration of TSS in the sample will be 23 mg/L. This means that for every 100 mL (0.1L) of sample used, the residue expected on the filter will be 2.3 mg. This equates to 0.0023g on the balance. Remember that the last

decimal place on your balance is considered the uncertainty of the balance. At lower residue weights a change of 1 on this last place can make a big difference. Let's assume that the level of TSS on the sample is at the lowest possible value of 23 mg/L. If you use 100 mL of sample, you would expect to see 0.0023g on the balance. A difference of 1 in the last decimal place is significant. If you take 0.0023 and divide by 0.0024 you get a difference of 4.2%. By contrast if you used 500 mL of sample, you would expect to see 0.0115g on the balance. A difference of 1 in the last decimal place becomes much less significant. If you take 0.0115 and divide by 0.0116 you get a difference of 0.9%.

- Never use a 2 place balance for measuring the residue. These balances are not sensitive enough to quantitate these low weights.
- Make sure you analyze a blank filter.
- Make sure that the pans and filter paper are dried before aliquoting samples.
- Cool pans in a dessicator.
- Rinse both the filtration apparatus and the container used for pouring the sample into the filtration apparatus, at least three times.
- Use a 4 place (analytical) balance.
- Dry each sample twice to make sure that all the moisture was removed in the first drying.

EXAMPLE CALCULATION FOR TSS

Initial weight of pan + filter paper	3.5579g
Initial dry weight of pan + filter paper + residue	3.5805g
Redry weight of pan + filter paper + residue	3.5807g
Amount of sample used	250 mL (0.25 L)

Calculation

$$\frac{(\text{Redry residue weight} - \text{initial weight of pan + filter})}{\text{Volume of sample used}} \times \frac{1000 \text{ mg}}{\text{g}}$$

$$\frac{(3.5807\text{g} - 3.5579\text{g})}{0.25\text{L}} \times \frac{1000 \text{ mg}}{\text{g}}$$

$$\frac{0.0228 \text{ g}}{0.25\text{L}} \times \frac{1000 \text{ mg}}{\text{g}}$$

91.2 mg/L

OIL AND GREASE ANALYSIS/SEPARATORY FUNNEL EXTRACTIONS.

- If possible, wear gloves. Your hands contain oils that if transferred to the weighing dishes can falsely add weight to the dishes.
- Dry your pans completely prior to aliquoting samples. It is recommended to use a light weight aluminum dish. The dish you use should be as light as possible. The heavier the dish weight, the more weight you will have to tare out when weighing residue.
- Dry the pans for at least one hour. Place the pans in a dessicator to cool to protect it from gaining moisture. Cool the pans for a minimum of 30 minutes. Obtain a weight of the empty pan
- Add the entire contents of the bottle into the separatory funnel. Rinse out the cap and the bottle both with the extraction solvent (usually hexane) three times and add the rinses to the funnel.
- Add 30 mL of hexane to the separatory funnel. Shake the funnel for 2 minutes to extract out the oil and grease into the solvent. Let the funnel settle to separate the water and hexane layers. Drain the water layer (bottom layer) back into the bottle. Then drain the hexane layer (top layer) into the pan.
- Repeat the above procedure two more times. Allow the hexane layer to evaporate in a fume hood. It is important to remove any water that is in the pan. The sample is made in tap water and may contain dissolved solids. When the water evaporates, the dissolved solids will be left in the pan which will falsely add weight to your residue.
- The water can be removed by immersing a disposable pipet into the sample and removing the water from the bottom of the pan.
- After the hexane has evaporated, place the pan into an oven at 70°C or less. Dry the sample for at least an hour. Remove the pan from the oven and place in the dessicator to cool. Cool for a minimum of 30 minutes. (It is advisable to cool for the same amount of time as was cooled before the initial weighing). Measure the weight of the dry and cool pan.
- Place the sample back into the oven for an additional 30 minutes. Place the sample back into the dessicator to cool. Reweigh the sample. This is to ensure that all moisture was removed during the initial drying period.
- Do not blank correct for grease and oil.
- Divide the gram weight measured for the residue by the volume of sample used (in liters) then multiply by 1000 to obtain a result in mg/L.

OIL AND GREASE ANALYSIS/AUTOMATED SOLID PHASE EXTRACTORS (SPE)

- Always refer to you instrument manufacturers operators manuals.
- Remove the cap and place the bottle on the extractor.
- Activate the filter disk by rinsing out with solvent (usually acetone or methanol). DO NOT USE HEXANE as this will cause the Oil and Grease material to pass through the disk prior to collecting the material passing through the filter into the collection vessel.
- Rinse off the cap of the bottle three times with hexane. Collect the hexane rinses in a separate container for recombination after the extraction procedure has been completed. Do not put the hexane rinses onto the activated disk as the hexane will solubilize the Oil and Grease and it will pass though the disk and go to waste.

- Begin the extractor and make sure that the filtrate goes to waste. All the grease and oil will collect on the extraction disk.
- Rinse the bottle three times with hexane and then collect these rinses into the collection vessel.
- After the hexane has evaporated, place the collection vessel into an oven at 70°C or less. Dry the sample for at least an hour. Remove the vessel from the oven and place in the dessicator to cool. Cool for a minimum of 30 minutes. (It is advisable to cool for the same amount of time as was cooled before the initial weighing). Measure the weight of the dry and cool pan.
- Place the sample back into the oven for an additional 30 minutes. Place the sample back into the dessicator to cool. Reweigh the sample. This is to ensure that all moisture was removed during the initial drying period.
- Do not blank correct for grease and oil.
- Divide the gram weight measured for the residue by the volume of sample used (in liters) then multiply by 1000 to obtain a result in mg/L.

TIPS AND TRICKS

- Run QC samples. These are samples that you know the certified value of and are provided with acceptance limits. For DMRQA they can be used as practice samples for you to determine how well your analysis is going. They are also great for use in your routine Quality Control program.
- 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 a dessicator.
- Make sure you remove any water from the collection pans. Water contains dissolved solids.
- Use a 4 place (analytical) balance.
- Dry each sample twice to make sure that all the moisture was removed in the first drying.

EXAMPLE CALCULATION FOR OIL AND GREASE

Initial weight of pan	13.5579g
Initial dry weight of pan + residue	13.5946g
Redry weight of pan + residue	13.5945g
Amount of sample used	1000 mL (1 L)

Calculation

$$\frac{(\text{Redry residue weight} - \text{initial weight of pan})}{\text{Volume of sample used}} \times \frac{1000 \text{ mg}}{\text{g}}$$
$$\frac{13.5945\text{g} - 13.5579\text{g}}{1 \text{ L}} \times \frac{1000 \text{ mg}}{\text{g}}$$
$$\frac{0.0366 \text{ g}}{1 \text{ L}} \times \frac{1000 \text{ mg}}{\text{g}}$$

36.6 mg/L

DEMAND/BOD AND CBOD ANALYSIS

PREPARATION OF THE ERA DEMAND CONCENTRATE

- If you are using ERA's Demand sample for your DMRQA BOD or CBOD analysis, the sample must be prepared ahead of time.
- Fill a 1 L volumetric flask with about 200 mL of distilled or DI water. Shake the Demand concentrate well. Using a glass pipet, remove 5 mL of the sample concentrate and add to the flask. Dilute to the line with water.
- The pH of the diluted sample will be around 3-4 s.u. Unlike your effluent samples which are already neutralized to the correct pH, the pH of the Demand sample must be adjusted to pH 6.5-7.5 s.u. If the pH is not adjusted the microorganisms needed for the oxygen depletion will not survive.
- It is recommended to adjust the pH in the following manner. Using a 0.2 N NaOH solution. Begin SLOWLY adding the NaOH to the flask. Continually measure the pH of the sample while adding the NaOH. Either add a stir bar to the flask and stir or manually shake the flask after each addition. After each addition, measure the pH. Continue adding NaOH slowly until the pH climbs into the acceptable range. It should take between 3-5 mL of 0.2 N NaOH to reach the desired pH. Do not use any more concentrated solution as the chances of overshooting the pH are great.
- BE PATIENT IT WILL TAKE SEVERAL MINUTES TO ADJUST THE pH. However, for successful analysis of this sample the pH needs to be right.

PREPARATION OF SOLUTIONS AND WATER

- Refer to Standard Method 5210 B for the BOD procedure.
- Prepare your nutrients for the nutrient water. There are four separate nutrients to add to the water. There is phosphate buffer, magnesium sulfate, calcium chloride, and ferric chloride. The nutrient water should be prepared by adding 1 mL of each one of the solutions per liter of dilution water. Alternately, commercially prepared nutrient solutions can be purchased (HACH Chemical Company).
- **Phosphate buffer:** Put about 500 mL of distilled or DI water in a 1L flask. Add 8.5g KH_2PO_4 , 21.75g K_2HPO_4 , 33.4g $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$, and 1.7g NH_4Cl to the flask. Dissolve and fill to the line with water.
- **Magnesium Sulfate solution:** Put about 500 mL of distilled or DI water in a 1L flask. Add 22.5g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ to the flask. Dissolve and fill to the line with water.
- **Ferric Chloride solution:** Put about 500 mL of distilled or DI water in a 1L flask. Add 0.25g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ to the flask. Dissolve and fill to the line with water.
- **Calcium Chloride solution:** Put about 500 mL of distilled or DI water in a 1L flask. Add 27g CaCl_2 to the flask. Dissolve and fill to the line with water.
- **Manganese Sulfate solution:** Put about 200 mL of distilled or DI water in a 1L flask. Add 480g $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$, or 400g $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$, or 364g $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ to the flask. Dissolve and fill to the line with water.
- **Alkali-iodide-azide reagent:** Put about 200 mL of distilled or DI water in a 1L flask. Add 500g NaOH (700g KOH) and 135g NaI (150g KI) to the flask. Dissolve and fill to the line with water. Add 10g NaN_3 dissolved in 40mL of water to the flask.

- **GGA standard:** Put about 500 mL of distilled or DI water in a 1L flask. Add 0.1500g glucose and 0.1500g Glutamic acid to the flask. Dissolve and fill to the line with water. This solution must be prepared fresh daily. This solution can also be made in a more concentrated form and diluted as needed. This solution can also be purchased commercially.
- **Sodium Sulfite solution:** Put about 500 mL of distilled or DI water in a 1L flask. Add 1.575g Na₂SO₃ to the flask. Dissolve and fill to the line with water. This solution must be prepared fresh daily. This solution is only needed to remove residual chlorine from the samples.
- **Nutrient water:** Purge the water to be used for the nutrient water with clean filtered air prior to using the water. This will saturate the water with dissolved oxygen. Add the nutrients to the water. This nutrient water mixture will usually be good only for about 24 hours. Measure the pH of the nutrient water. The pH must be between 6.5-7.5 s.u. Adjust the pH with either acid or base, if necessary.
- **Nitrification inhibitor:** It is recommended to purchase a commercially available nitrification inhibitor (2-chloro-6-(trichloromethyl) pyridine - TCMP).
- **Seed:** There are several different types of seeds that can be used. There is a commercially available seed. Most plant influents (and some effluents) make outstanding seed. The easiest way to prepare the seed is to collect some influent. Allow the influent to stand for 1-24 hours. Decant of the supernatant liquid and use this for your seed. You can also filter the influent through a 0.75µm filter paper.
- It is recommended to add seed directly to the BOD bottles and not to the nutrient water.

PREPARING/ANALYZING THE SAMPLES USING A MEMBRANE PROBE

- Put nitrification inhibitor into the BOD bottle if performing CBOD. Add an appropriate amount of sample to the BOD bottle. Add seed to the bottle. Fill the remaining 300mL of the BOD bottle with nutrient water. The bottle should be full right up to the base of the neck of the bottle. Do several dilutions for the samples. Based on historical knowledge of DO depletion for known concentrations (the GGA standard is a good reference) make dilutions appropriate for a BOD of 15-250 mg/L.
- Set-up your dissolved oxygen probe. Allow the probe to warm up prior to making any readings. It is highly recommended to change the membrane on the probe routinely. (Usually at least once a week). Also it is highly recommended to score the gold sensor at the bottom of the probe routinely to remove any buildup. (Usually once a month will suffice).
- Worn membranes or buildup on the probe can cause the probe to drift significantly.
- Calibrate the DO probe every day of use. The best way to calibrate the probe is to check the DO of the water on each day by using the Winkler titration method. The DO probe should then give the same DO reading as was obtained using the titration method. The calibration of the probe should be checked periodically during the analysis of the BOD bottles.
- Stir the BOD bottle with the DO probe. Take an initial DO reading. Place the cap on the bottle. There should be a small amount of fluid on the outside of the bottle where the cap is. This fluid is critical in this analysis as it prevents oxygen from entering the BOD bottle during incubation. Place some aluminum foil or a small plastic cup over the BOD

bottle cap to prevent the fluid on the outside of the bottle from drying out during incubation.

- Incubate the sample in a constant temperature incubator at $20 \pm 1^\circ\text{C}$ for 5 days.
- Remove the sample from the incubator after 5 days. Calibrate the DO probe the same way as prior to incubation. Take final DO readings.
- Calculate the BOD as detailed below.

WINKLER TITRATIONS FOR DETERMINING DISSOLVED OXYGEN (METHOD 4500-O C)

- Preparation and incubation of the samples is performed the same as for the membrane probe method, with the exception of preparing two bottles of each sample as opposed to just one bottle.
- Determine the initial DO of the sample. Add 1 mL of the MnSO_4 solution prepared above. Then add 1 mL of alkali-iodide-azide reagent. Place the stopper on the bottle carefully to not allow air bubbles into the bottle. Invert the sample several times. Allow the sample to settle until there is at least half of the bottle of supernatant liquid. Add 1 mL of sulfuric acid. Restopper the bottle and invert several times. When dissolution is complete, take out 200 mL of sample and place in a 250 mL beaker. Place a stir bar in the sample and place the sample onto a stir plate.
- Titrate the sample with standardized 0.025M sodium thiosulfate solution. Until it reaches a pale straw color. Add a few drops of a starch indicator. The solution will turn blue. Titrate slowly until the blue color disappears. For 200 mL of sample, 1 mL of 0.025M sodium thiosulfate solution is equal to 1 mg/L of DO.
- Incubate the second bottle in a constant temperature incubator at $20 \pm 1^\circ\text{C}$ for 5 days.
- Remove the sample from the incubator after 5 days. Take final DO readings using the same procedure as the initial DO readings.
- Calculate the BOD as detailed below.

TIPS AND TRICKS

- Cleanliness is next to godliness. Keep everything used for this test clean. Residual chlorine is a major interferent. It will kill the microorganisms used in the test. Make sure that any chlorine is removed from the water, glassware, and any reagents used for this test.
- Nitrifying bacteria is an interferent in CBOD analysis. Be cautious using plant influent as a seed if a high percentage of the bacteria in the seed is nitrifying bacteria. The nitrification inhibitor will prevent any nitrifying bacteria from depleting the dissolved oxygen and it may not leave you with enough carbonaceous bacteria to generate a proper oxygen depletion.
- Make sure the incubator temperature remains at $20 \pm 1^\circ\text{C}$. A higher temperature will increase the aerobic activity and thus increase the oxygen depletion. A lower temperature will decrease aerobic activity and thus decrease aerobic activity.
- Make sure to subtract out the value per mL of the seed control from the oxygen depletion value prior to calculating the BOD value.

- Analyze a seeded and an unseeded blank to make sure that either the seed or water aren't introducing BOD contamination into your process.
- Initial DO readings should be 7-9 mg/L. If the initial DO is greater than 9, the sample is supersaturated. Agitate the sample by shaking or aerating to bring the DO to 7-9 mg/L
- Final DO readings (residual DO) greater than 1.0 mg/L
- The DO depletion should be at least 2.0 mg/L
- Keep samples out of the light as much as possible. Light can trigger production of DO.
- The seed control process should obtain a DO uptake of the seed somewhere at 0.6-1.0 mg/L.
- Calculate the DO uptake of the seed per mL of seed used. This value must be subtracted out from the total DO uptake prior to making calculations.
- Try to use the same amount of seed for all samples.

EXAMPLE CALCULATIONS

Seed Control DO uptake

$$SC = \frac{(S_1 - S_2)}{V}$$

Where:

S_1 = Initial DO reading of seed control, in mg/L

S_2 = Final DO reading of seed control, in mg/L

V = volume of seed used, in mL

BOD Calculation

If the seed control was not calculated per mL of seed

$$BOD_5, \text{ mg/L}, = \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$

Or if the seed control was calculated per mL of seed

$$BOD_5, \text{ mg/L}, = \frac{(D_1 - D_2) - (SC)V}{P}$$

Where:

D_1 = Initial sample DO reading, in mg/L

D_2 = Final sample DO reading after incubation, in mg/L

B_1 = Initial seed control DO reading, in mg/L

B_2 = Final seed control DO reading, in mg/L

f = (volume of seed in sample)/(volume of seed in seed control)

P = Decimal fraction of sample used

SC = DO uptake of the seed control, in mg/L per mL of seed

V = volume of seed used, in mL

pH ANALYSIS

- Calibrate the pH meter.
- It is recommended to screen the pH sample first. This will give you a rough idea of what the pH will be.
- It is recommended to calibrate the pH meter with two buffer solutions. Depending on the results of the screen, calibrate the meter with either pH buffers 4 and 7 or pH buffers 7 and 10.
- pH buffers are available very inexpensively from commercial vendors.
- Ensure that all solutions run for pH are at room temperature. Most pH meters are equipped with ATC (Automatic Temperature Compensation) units. However, if the temperature difference between the buffers and the samples is too great the ATC is rendered ineffective.
- Follow the manufacturer's instructions for a two buffer calibration.
- Shake the sample well before aliquoting for pH analysis.
- Allow the samples to equilibrate on the meter.

TIPS AND TRICKS

- Run QC samples. Using a secondary source for pH will help verify that the pH buffers are still good.
- Always run buffers and samples at room temperature.
- Use a stirring plate and stir bar with pH analysis. The sample being measured needs to be mixed very well.
- Analyzing samples for pH that are not at the same temperature can cause varying results. Always, if possible, use an ATC unit. It will correct your pH results for differences in temperature.
- Always use fresh buffers. When the buffers and samples are exposed to air, they are very susceptible to carbon dioxide (CO₂) adsorption. CO₂ adsorption will drop the pH of the buffers or samples. Therefore, if the buffers or samples are reused over time, the pH of each will change.
- Always let the meter equilibrate.
- Do not calibrate with either one buffer or all three buffers. One buffer does not provide enough accuracy at any end of the range. Using three buffers may allow you to run samples throughout the range but will decrease accuracy overall.

TOTAL RESIDUAL CHLORINE ANALYSIS

PREPARATION OF THE ERA TOTAL RESIDUAL CHLORINE CONCENTRATE

- If you are using ERA's Total Residual Chlorine sample for your DMRQA Total Residual Chlorine analysis, the sample must be prepared ahead of time.
- Fill a 1 L volumetric flask with about 200 mL of distilled or DI water. Shake the Total Residual Chlorine ampule well. Snap the top off of the ampule. Using a glass pipet or a gastight syringe, remove 1 mL of the sample concentrate and add to the flask. Dilute to the line with water.

TOTAL RESIDUAL CHLORINE ANALYSIS/SPECTROPHOTOMETER

- As Chlorine is unstable, you should analyze the sample as soon as possible. It is best generally to analyze the sample with about 2 hours after diluting the sample.
- If you are using a HACH spectrophotometer, follow the HACH instructions for analysis. Make sure you are using the appropriate powder pillow for the amount of sample and meter being used for the analysis.
- Zero out the instrument using just sample with no powder pillow added. This will remove any bias from the natural coloring of the sample.
- Run a QC sample as designed by HACH to check your instrument.
- Allow the color to develop fully. This usually takes a few minutes.
- If the instrument is saturated, the readout on the panel should blink. If this happens, it means the concentration of the sample is too high. The sample must be further diluted. Try doing a 2 fold dilution first. This should be all it needs. If you are using a 10 mL sample volume, take 5 mL of the sample and 5 mL of the dilution water and mix them in the sample sup. Add the powder pillow and analyze. Remember that only half of what you are analyzing is sample. Thus when you report your results you must multiply the final number by two.

TIPS AND TRICKS

- Make sure all your glassware is clean and free from any chlorine.
- Analyze the sample ASAP as chlorine degrades quickly.
- Run QC samples.
- Make sure your reagents are fresh. Commercially available reagent packets are great for this test.
- Make sure the powder pillow you are using is appropriate for the amount of sample you are using.
- Glass pipets from Kimble have a long skinny nose on the end and fit into the chlorine ampule perfectly. The glass pipets from pyrex have shorter and fatter noses and do not fit as well. If possible, it is recommended to use a 1 mL gastight syringe.

SIMPLE NUTRIENTS/AMMONIA AS N

PREPARATION OF THE ERA SIMPLE NUTRIENTS CONCENTRATE

- If you are using ERA's Simple Nutrients sample for your DMRQA Ammonia as N analysis, the sample must be prepared ahead of time.
- Fill a 1 L volumetric flask with about 200 mL of distilled or DI water. Shake the Simple Nutrients concentrate well. Using a glass pipet, remove 5 mL of the sample concentrate and add to the flask. Dilute to the line with water.
- Distillation of this sample is not required for this sample. The ammonia is already free in solution. Once diluted, the sample is ready for analysis.

AMMONIA AS N ANALYSIS/ION SELECTIVE ELECTRODE

- Prior to analysis, the electrode must be prepared. The electrode contains a gas permeable membrane that must be periodically changed. To change the membrane, unscrew the bottom of the electrode. Remove the old membrane and drain out the filling solution. Place a new membrane over the whole in the bottom of the outer electrode body. Make sure you wear gloves when changing the membrane. The oils on your hand will clog the membrane. Fill the inside of the outer body with ammonium chloride filling solution. Place the inner electrode inside the outer body. If the electrode was dry it is recommended to let the electrode condition overnight. If the electrode was already filled with solution and was just changed, then 1 hour of conditioning should suffice.
- Allow the electrode to warm up for about an hour prior to analysis. Add 100 mL of DI water to a 150 mL beaker. Add 2 mL of ISA to the solution. Add a stir bar to the beaker and stir the sample with a stirring plate. Immerse the electrode into the solution.
- Prior to analysis of samples a slope of the electrode must be performed. The slope of the electrode is performed to ensure that the electrode is responding properly. Proper response of the electrode is shown by a change in the mV readings of 57 mV for every 10 fold change in concentration. Most electrodes have an acceptable range for the mV change of 54-60 mV.
- To perform the slope, use a 100 mg/L or 1000 mg/L ammonia solution. Add 1 mL of sample to the beaker. Allow the mV readings to stabilize and record the reading. Then add an additional 10 mL of the same ammonia solution to the beaker and allow the mV readings to stabilize. Record the mV reading. Subtract the second mV reading from the first. The change should be 54-60 mV. If the value is outside of this range then the electrode operation must be evaluated and corrected.
- Once the slope is completed, an initial calibration must be performed. The concentration of the initial calibration standards must be chosen to ensure that the electrode can accurately quantitate at the lower end and not saturate at the upper end. All samples analyzed should be bracketed by this calibration curve to ensure maximum accuracy.
- Most meters can be programmed to display results in concentration and mV. For either setup, make sure that the readings are stable for the calibration standards before instructing the meter to continue.

- If the meter reads out in mV, calculations must be performed to convert the meter readings to concentration.
- For both the calibration standards and samples, use 100 mL of sample. For ammonia analysis, 2 mL of Ionic Strength Adjustor (ISA) should be adequate. If using NaOH to adjust pH, the pH must be adjusted and monitored greater than 11 for the ammonia to be released. ISA solution is colored and will remain that color when the correct pH is attained. Stir the sample until either a constant mV or concentration is attained.
- Temperature plays a big part in the ammonia analysis. It is important to make sure that all analyses are performed at a constant temperature. A 1° change in temperature can cause up to a 5% error. The stirring plate can also cause heat transfer to the sample. Place something on top of the hotplate to prevent heat transfer from the plate to the sample. Corkboard or packing material works great.
- Run calibration checks during the analysis. The meters have a tendency to drift.

TIPS AND TRICKS

- ANALYZE AND REPORT YOUR AMMONIA RESULTS AS N AND NOT NH₃.
- Run QC samples.
- Make sure the membranes are changed routinely. Usually once per week.
- Calibrate the meter each time you analyze samples. Keep the dynamic range of the curve as small as possible to encompass the expected concentration of the samples to be run. The wider the curve the less accurate your quantitation will be.
- Use a colored Ionic Strength Adjustor (ISA). Orion makes a blue one. When the ISA is added to the sample, the sample will remain blue when the pH is greater than 11.
- Make sure stock standards are fresh.
- Run QC checks during analysis.
- Keep temperature as constant as possible.



DMR-QA 30 Data Reporting Cover Sheet

Lab Name: ERA

Customer Number: E667501

A) LABORATORY INFORMATION

Below is the information we currently have on file for your laboratory. Please note that the address shown below is where your final report will be sent. If there are any corrections, please fill in the boxes below the appropriate heading.

Lab Name: ERA

Mailing Address: 6000 W. 54th Ave.

City: Arvada

ST: CO

Email Address: acornelli@eraqc.com

USEPA ID: CO00000

Phone: 303-431-8454

 - -

Fax: 303-421-0159

 - -

Zip: 80002

 -

B) Where would you like ERA to send your Final report?

By participating in ERA's Proficiency Testing Study, you will automatically receive a Final Report approximately 21 days after the study closes. To authorize ERA to send a report to your accrediting authority, please fill in the oval to the left of the accrediting authority(ies) to whom you wish ERA to send your Final Report.

	State ID		State ID		State ID		State ID
<input type="checkbox"/> Iowa	✓ / ✓		<input type="checkbox"/> North Carolina (WP)	✓ / ✓		<input type="checkbox"/> South Carolina	✓ / ✓
<input type="checkbox"/> Kansas	✓ / ✓		<input type="checkbox"/> North Dakota	✓ / ✓		<input type="checkbox"/> Virginia (WP)	✓ / ✓
						<input type="checkbox"/> Washington	✓ / ✓

The agencies listed here will accept DMR-QA for state waste water or WP accreditation. Only select one of these states if you wish to report data for state WP accreditation.





A Waters Company

DMR-QA 30 Data Reporting Cover Sheet

Lab Name: ERA

Customer Number: E667501

C) Enter OTHER ENTITIES

In addition to sending your report to your accrediting authority, you may authorize ERA to send copies of your report to other entities (e.g. customers, corporate QA officers, etc.) by simply filling in the information below. If you need ERA to submit your final report to more than two third parties, please photocopy this form.

Company:

Attention:

Mailing Address:

City:

ST:

Zip:

Company:

Attention:

Mailing Address:

City:

ST:

Zip:

D) Read and sign the ATTESTATION STATEMENT

Per the requirements of the USEPA's National Standards Criteria Document, please read this attestation statement. By affixing your signature below, you attest that the results have met the following criteria: 1) No results, or any other aspect of the study, have been revealed to or discussed with any unauthorized person or other laboratory prior to the close of the study. 2) The standards for which you are submitting results were not analyzed by any other laboratory. 3) Your laboratory has not knowingly received standards from any other laboratories. 4) No information was solicited from ERA or any other laboratories concerning the assigned values or acceptance ranges for the standards until the close of the study. 5) All analyses met the criteria for the regulatory agencies to which the results are being sent.

Official Laboratory Signature: _____ Date: _____

Official Laboratory Title: _____ Phone: _____

Printed Name of Signator: _____

- Return the Data Reporting Cover Sheets (2) plus your "DMR-QA 30 DATA REPORTING FORM(S)" to ERA by Fax or Mail.
- ERA's fax number is 1-720-898-6382
- Total pages: _____
- Questions? See the DMR-QA 30 Data Reporting Instructions or call ERA at 1-800-372-0122.





A Waters Company

DMR-QA 30 Data Reporting Form

NPDES Permit #:

Permittee Name:

Lab Name: ERA

ERA Customer Number: E667501

USEPA Lab Code: C000000

INSTRUCTIONS:

Please fill in the results, methods references and analysis dates for the analyte(s) you wish to report for the DMR-QA 30 study. Questions? See the Data Reporting Instructions section of your Data Package or call ERA at 1-800-372-0122. Please photocopy this form if you are reporting multiple methods

DMR-QA Minerals (cat# 581)

Anal No.	Analyte	Units	Reported Value	Method Description	Analysis Date (mm-dd)	Rpt* For DMR
0027	Alkalinity as CaCO3	mg/L	.		-	<input type="checkbox"/>
0028	Chloride	mg/L	.		-	<input type="checkbox"/>
0020	Conductivity at 25°C	umhos/cm	.		-	<input type="checkbox"/>
0029	Fluoride	mg/L	.		-	<input type="checkbox"/>
0026	Potassium	mg/L	.		-	<input type="checkbox"/>
0025	Sodium	mg/L	.		-	<input type="checkbox"/>
0030	Sulfate	mg/L	.		-	<input type="checkbox"/>
0021	Total Dissolved Solids at 180°C	mg/L	.		-	<input type="checkbox"/>
1950	Total Solids at 105°C	mg/L	.		-	<input type="checkbox"/>


*For Permittee use only:

Place an "X" in the box to the right of each analyte that is required in routine NPDES monitoring and should be included on the Final Permittee DMR-QA 30 report.

Optional section. Please use to specify Analyst and Work Group.

Anal No.	Analyte	PTRL	Concentration Range	Analyst	Work Group
0027	Alkalinity as CaCO3	6.80	10.0 to 120		
0028	Chloride	29.0	35.0 to 275		
0020	Conductivity at 25°C	170	200 to 930		
0029	Fluoride	0.130	0.300 to 4.00		
0026	Potassium	3.00	4.00 to 40.0		
0025	Sodium	5.10	6.00 to 100		
0030	Sulfate	2.80	5.00 to 125		
0021	Total Dissolved Solids at 180°C	98.0	140 to 650		
1950	Total Solids at 105°C	106	140 to 675		



	<p>United States ENVIRONMENTAL PROTECTION AGENCY Washington, DC 20460 Laboratory DMR-QA Evaluation Study 31 Laboratory Performance Evaluation Office of Enforcement and Compliance Assurance <small>(These data are collected under the authority of the Federal Water Pollution Control Act.)</small></p>
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NPDES Permittee Data Report Form

Due August 19, 2011

Attention: Follow the instructions on the previous page to complete this form and submit data for evaluation.

State	NPDES Permit Number	Permit Extension
<input type="text"/>	<input type="text"/>	<input type="text"/>

Permittee name

Current Permittee mailing address

City State Zip Code

Phone Number FAX Number

e-Mail

For DMR-QA Study 31, conducted in 2011, the Permittee ensured that their laboratory(s) performing the required analyses:

Received PT Samples	Submitted Complete and Accurate Data by July 1, 2011	Received a Graded Report by July 22, 2011
Yes <input type="checkbox"/> No <input type="checkbox"/>	Yes <input type="checkbox"/> No <input type="checkbox"/>	Yes <input type="checkbox"/> No <input type="checkbox"/>

Certification by Permit Holder or Authorized Representative
(as per 40 C.F.R. Section 122.22 - see instructions.)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. Each reported value was produced from a single analytical run using the analytical system that routinely performs these analyses to produce compliance monitoring data required under our National Pollutant Discharge Elimination System (NPDES) permit. Neither I nor any of my subordinates compared our results with results from independent analyses conducted by us or any other laboratory before we reported our results to the U.S.EPA. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.


Name of Certifying Official Title

Signature _____ Date

Address, phone number and e-mail of certifying official are required if different from above.

Address Phone No.

City State Zip Code E-mail

	United States ENVIRONMENTAL PROTECTION AGENCY Washington, DC 20460 Laboratory DMR-QA Evaluation Study 31 Laboratory Performance Evaluation Office of Enforcement and Compliance Assurance (These data are collected under the authority of the Federal Water Pollution Control Act.)
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Permittee name <input style="width: 95%;" type="text"/>	State <input style="width: 95%;" type="text"/>	NPDES Permit No. <input style="width: 95%;" type="text"/>	Permit Extension <input style="width: 95%;" type="text"/>
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Identification of all CHEM, MICRO and WET laboratories who did analyses for this permit							
Name of Laboratory	Address of Laboratory	U.S. EPA Lab Code	Lab Analysis Check box(es) that apply			Lab Type*	State-certified Laboratory**
			Chem	Micro	WET		
<input style="width: 95%;" type="text"/>	<input style="width: 95%;" type="text"/>	<input style="width: 95%;" type="text"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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<input style="width: 95%;" type="text"/>	<input style="width: 95%;" type="text"/>	<input style="width: 95%;" type="text"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

* Lab Types: C = Commercial F = Federal G = Local Government I = Industrial O = Other S = State
 ** See Footnote 2 on DMR-QA Study 31 Fact Sheet

If you need additional space, please make a copy of this page for additional laboratories.

Permittee name _____ State _____ NPDES Permit No. _____ EPA Lab Code _____

Chemistry/Microbiology Analyte Checklist
DMR-QA Study 31

Analyte Test	Test Required	Laboratory's Graded Result		Analyte determined by state-certified laboratory*
		Acceptable	Not Acceptable (Corrective Action Required)	
Microbiology				
E. coli	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Fecal Coliform, MF or MPN	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Coliform, MF or MPN	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Trace Metals				
Aluminum	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Antimony	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Arsenic	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Barium	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Beryllium	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Cadmium	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Chromium, total	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Chromium, hexavalent	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Cobalt	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Copper	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Iron	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Lead	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Manganese	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Mercury	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Mercury (Low Level)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Molybdenum	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Nickel	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Selenium	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Silver	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Thallium	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Vanadium	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Zinc	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Demands				
5-day BOD	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
5-day Carbonaceous BOD	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
COD	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
TOD	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Minerals				
Alkalinity, total (CaCO ₃)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Chloride	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Fluoride	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Hardness, total (CaCO ₃)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Specific conductance (25°C)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sulfate	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Dissolved Solids (180°C)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Nutrients				
Ammonia as N	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Nitrate as N	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Nitrite as N	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Orthophosphate as P	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Kjeldahl-Nitrogen as N	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Phosphorus as P	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Misc. Analytes				
Non-Filterable Residue (TSS)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Oil and Grease	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
pH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Cyanide	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Phenolics (4-AAP)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Residual Chlorine	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Residual Chlorine (Low Level)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Settleable Solids	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Turbidity	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Signature _____ Date _____

*See Footnote 2 on DMR-QA Study 31 Fact Sheet

Use a separate checklist for EACH lab used

Permittee name _____ State _____ NPDES Permit No. _____ EPA Lab Code _____

WET Organisms/Test Conditions/End Points Checklist

DMR-QA Study 31

Analyte Number	Organisms / Conditions	End Points	Test Required	Laboratory's Graded Result		Analyte determined by state-certified laboratory*
				Acceptable	Not Acceptable (Corrective Action Required)	
Test Code 13/EPA Method 2000						
754	Fathead minnow (<i>Pimephales promelas</i>) - MHSF 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 14/EPA Method 2000						
755	Fathead minnow (<i>Pimephales promelas</i>) - 20% DMW	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 15/EPA Method 1000						
756	Fathead minnow (<i>Pimephales promelas</i>) - MHSF	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
808	Fathead minnow (<i>Pimephales promelas</i>) - MHSF	IC25 (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
810	Fathead minnow (<i>Pimephales promelas</i>) - MHSF	NOEC (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 16/EPA Method 1000						
759	Fathead minnow (<i>Pimephales promelas</i>) - 20% DMW	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
812	Fathead minnow (<i>Pimephales promelas</i>) - 20% DMW	IC25 (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
814	Fathead minnow (<i>Pimephales promelas</i>) - 20% DMW	NOEC (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 19/EPA Method 2002						
764	<i>Ceriodaphnia dubia</i> - MHSF 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 20/EPA Method 2002						
765	<i>Ceriodaphnia dubia</i> - 20% DMW 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 21/EPA Method 1002						
766	<i>Ceriodaphnia dubia</i> - MHSF	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
767	<i>Ceriodaphnia dubia</i> - MHSF	IC25 REPRODUCTION	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
768	<i>Ceriodaphnia dubia</i> - MHSF	NOEC REPRODUCTION	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 22/EPA Method 1002						
769	<i>Ceriodaphnia dubia</i> - 20% DMW	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
770	<i>Ceriodaphnia dubia</i> - 20% DMW	IC25 REPRODUCTION	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
771	<i>Ceriodaphnia dubia</i> - 20% DMW	NOEC REPRODUCTION	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 32/EPA Method 2021						
788	<i>Daphnia magna</i> - MHSF 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 38/EPA Method 2021						
794	<i>Daphnia pulex</i> - MHSF 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 42/EPA Method 2007						
798	<i>Mysid (Mysidopsis bahia)</i> 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 43/EPA Method 1007						
799	<i>Mysid (Mysidopsis bahia)</i>	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
816	<i>Mysid (Mysidopsis bahia)</i>	IC25 (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
818	<i>Mysid (Mysidopsis bahia)</i>	NOEC (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 44/EPA Method 2006						
803	Inland silverside (<i>Menidia beryllina</i>) 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 45/EPA Method 1006						
824	Inland silverside (<i>Menidia beryllina</i>)	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
825	Inland silverside (<i>Menidia beryllina</i>)	IC25 (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
826	Inland silverside (<i>Menidia beryllina</i>)	NOEC (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 46/EPA Method 2004						
804	Sheepshead minnow (<i>Cyprinodon variegatus</i>) 25°C	LC50	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Test Code 47/EPA Method 1004						
805	Sheepshead minnow (<i>Cyprinodon variegatus</i>)	NOEC SURVIVAL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
820	Sheepshead minnow (<i>Cyprinodon variegatus</i>)	IC25 (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
822	Sheepshead minnow (<i>Cyprinodon variegatus</i>)	NOEC (ON) GROWTH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Name _____ Signature _____ Date _____

*See Footnote 2 on DMR-QA Study 31 Fact Sheet