

The Pipeline

Drinking Water Laboratory
Policy & Procedures Update

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ALERT!: Contaminated Colisure in Glass Vessels

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This notice is only for laboratories that have used the lyophilized form of Colisure (supplied in 125 mL glass test vessels), during 1997. This should only apply to a very small percentage of laboratories in Ohio. This does not apply to any lots of granulated Colisure reagent supplied in small screw capped vials or the latest Colisure manufactured by Idexx, supplied in snap packs. This office has been supplied with a document from Millipore Corporation, the former supplier of Colisure MMO-MUG test reagent, that notifies of three contaminated lots of Colisure reagent. The lots affected are Millipore Colisure Presence/Absence Lyophilized Reagent in Glass test Vessels, catalog number MC000PA00, lot numbers: 7215; 7230 and 7240. Since the contamination occurred quite some time ago (we were only made aware of this in October 1998), you should not have any of these lots left. If you do, return them to the manufacturer. The contamination was low level and only affected a small percentage of each lot. However, please go through your records and see if you have used these lots in the past. **If you have used them and generated positive results with them, the positive results must be invalidated.** Please note this in your records and notify your Ohio EPA district office. **If you analyzed tests for another entity or are a commercial lab, you must notify all customers that the positive test results are invalid.** Evidence of such notification must be available upon request during an on-site survey. Negative test results can still be used. Please note that Millipore discontinued this form of product (lyophilized, in glass test vessels) shortly after the problem was discovered. Millipore no longer supplies Colisure, Idexx Corporation now handles Colisure. There have been no contamination problems reported since this incident.

PE/WS Update



As many of you know the USEPA "WS" program has ended. The last set of samples has already been sent. In the future, Ohio will have to plan and manage the drinking water performance evaluation (PE) sample program. PE's will be required for all chemistry tests with an MCL with the exception of: turbidity and fluoride. Fluoride PE samples will be required for commercial laboratories. Water plants participate in the monthly split sample program for fluoride, so a PE is not required. Tests which have no MCL such as: alkalinity, pH, stability, hardness, chloride and chlorine will not require PE samples. Chemistry PE tests will be required at least twice each year. Laboratories certified for microbiology procedures will also be required to participate in the PE program, at least once each year. It is anticipated that The Ohio EPA will approve a PE supplier in the near future. Laboratories will be given the name and address of the approved PE supplier and will be required to use only that facility for PE samples. Each certified laboratory will be required to pay for their own samples. The Ohio EPA should draft a procedure for PE tests, including what happens when you miss a sample within the next several months. The program should begin in the winter or spring of 1999.

High Level Chlorine Test Notes

If your water plant tests chlorine levels of 2.0mg/L or higher, you should be sure you are using the correct procedures for chlorine and alkalinity tests.



As noted in past Pipelines, for chlorine levels of 2.0 mg/L or higher, use two, 10mL packets of Hach DPD reagent for each 10mL test, or one 25mL test packet for each 10mL test. This applies to any colorimetric test instrument that use powdered DPD reagents, including spectrophotometers (which includes DR-100's). This also applies to both free and total chlorine determinations. If your instrument uses a 25mL test sample, then use two 25mL packets. In other words, if you see chlorine levels of 2.0mg/L or more, double the dose. If you use a DPD powder dispenser, use two doses for levels of chlorine 2.0mg/L or greater.

When the chlorine level approaches 2.0 mg/L or higher it can also affect the alkalinity test. In this case add one or two drops of a 10% sodium thiosulfate solution to neutralize the chlorine before adding the indicator to the test vessel. You may notice poor color change at chlorine levels as low as 1.5 mg/L. In this case, you may add the sodium thiosulfate to the sample also.

Change in Reagent Storage Times

The following are changes in the official policy concerning storage time limits for test materials: Single use DPD powder packets may now be used until the manufacturer's expiration date, which is printed on each packet, is reached. Hach brand chlorine ampules, used for calibrating chlorine test instruments may be kept for two years after receipt, in the refrigerator.

These changes supersede requirements noted in the chemical certification manual. Please transfer these changes to your manual.

Using Micropipettors

Many laboratories have been observed using micropipettors incorrectly on surveys lately. Different brands of micropipettors vary in proper use. You are urged to get out the manufacturer's instructions and read them as to the proper use. Some general usage notes that may apply to most brands are:



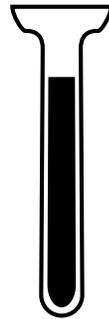
Usually, you should start with the lowest spike when doing multiple spikes, then work your way up to the largest spike.

It isn't necessary to change tips when measuring from the same standard, if you start at the lowest and work your way to the highest spike.

When adjusting to the volume mark, most manufacturers' require always adjusting down to the volume mark. In other words when spiking a 50uL and the a 75uL, when adjusting from 50 to 75, dial up to 85 then back down to 75. This takes the mechanical lash out of the instrument.

Use the micropipettor plunger smoothly and not too fast.

Before withdrawing the test portion, press the plunger to the first stop out of the liquid, not all the way to the bottom, place the tip in the liquid, withdraw, expel the liquid by pressing all the way to the second stop. Some pipettors do not have the second blow-out stop. Consult the manufacturer's instructions in these cases.



Turbidity Updates

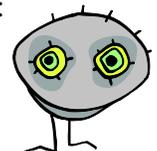
When using the Hach 2100N/AN Turbidimeter, many labs have been performing and recording a daily check using 0-2 NTU, 0-20 NTU and a 0-200 NTU secondary standards. The only required standards are the 0-2 NTU and 0-20 NTU secondary standards. You are not required to use the 0-200 NTU standards for daily checks. Remember: for 20 NTU and lower, the ratio switch must be off. "Signal averaging" may be used for all tests.

If Hach StablCal primary standards or AMCO primary standards are used in your laboratory, they must be bulk standards and not sealed ampuled standards. Ampuled StablCal and Ampuled AMCO standards are meant to be secondary standards, not primary standards. A primary standard is to be used, then discarded, with a new portion used for each standardization. Both Hach and AMCO sealed ampules are acceptable for use as secondary standards after they have been calibrated to their true value with the primary standards.

Hach StablCal standards come with specific instructions concerning their proper use. This includes the proper agitation instructions. Follow the manufacturer's requirements for preparation.

Microbiology New Test Information

Many laboratories are probably aware that the USEPA has approved several new methods for total coliforms. One of them is the 18 hour MMO-MUG test. Since Ohio has primary enforcement responsibility for the drinking water programs, we can choose to be more stringent than USEPA. This also means that we can limit the number of tests used by laboratories within Ohio. Currently the only tests approved for use in Ohio are the m-Endo, membrane filter test and 24 and 28 hour MMO-MUG tests (Colilert and Colisure). We hope to evaluate some of these new tests soon. The evaluations, hopefully, will be conducted by certified laboratory personnel through the AWWA Research Committee. If you may be interested in participating in such research, please contact Don LaSota at (614) 644-4266 or E-mail: don.lasota@epa.state.oh.us



UPDATE: Buffered Rinse Water for MF

The 09-03-98 (Volume 63, Number 171) Federal Register contained a notice that the USEPA is proposing to approve the updated versions of *Standard Methods for the Examination of Water & Wastewater* and the Environmental Protection Agency analytical methods for compliance determinations of microbiological contaminants in drinking water. A direct result of this action is that the Ohio EPA may no longer continue to use the optional formulation of buffered rinse water that has been used in the past. The new formulation is similar to the old formulation with the addition of a magnesium chloride stock solution to the final buffered rinse water. The addition of the magnesium chloride stock solution is supposed to enhance the recovery of some stressed organisms according to some studies. To give the laboratories time to order supplies and prepare the new formulation of buffered rinse water, the Laboratory Certification Section will not enforce this change until April 1, 1999. During the next six months laboratories approved for the membrane filter procedure may make the change as the materials are received.

Buffered Rinse Water (Revised 10-98)

Add 1.25 mL of stock buffer solution and 5.0 mL magnesium chloride stock solution (preparations to follow) per liter of laboratory pure water, to be used as rinse water for the membrane filtration procedure. Allow the stock buffer and $MgCl_2$ solutions to come to room temperature in order to accurately measure the volume. Use a 2.00 mL pipet (calibrated in 1/100's) to deliver 1.25 mL, and use a 5.0 mL pipet to deliver 5 mL. Use a one liter graduated cylinder, or a one liter volumetric flask to measure one liter of laboratory pure water. If larger volumes are prepared, it is permissible to mark the rinse water container at the calibrated volume level with a permanent mark. Sterilize the rinse water as soon as the buffer has been added. Do not store the stock buffer, $MgCl_2$, distilled water solution in non-sterile condition for >4 hours. Store sterile rinse water for up to six months at room temperature. Do not refrigerate the rinse water.

Stock phosphate buffer with a pH of 7.2 ± 0.2 is prepared as follows: Dissolve 34 G of monobasic potassium phosphate in 500 mL of laboratory pure water in a 1 liter volumetric flask. Do not use phosphate buffer used for BOD analysis because it has a different composition. After dissolving the reagent; adjust the pH to 7.2 ± 0.2 with 1.0 or 0.1N NaOH. Let the solution stand for a few hours. Recheck the pH; note initial pH. Dilute the volume to 1000 mL. Optionally, "BBL APHA Phosphate Buffer 7.2" may also be used. Follow the manufacturer's instructions for preparation. The pH of this solution is self adjusting and cannot be adjusted before or after autoclaving. Dispense stock buffer solution in 10 to 20 mL volumes in screw cap tubes or vessels. Autoclave for 15 minutes at 119 - 121% C. The pH will generally drop during sterilization. Check and record final pH when cool. If the final pH is not 7.2 ± 0.2 , discard it and prepare a second batch. If you are not sacrificing a complete container for the final pH reading, use a sterile pipet to remove the pH portion, do not put the electrode into the sterile solution.

Date each batch of stock buffer with the date of preparation. Store in the refrigerator at 0.0 - 5.0% C for not more than six months. Prepare fresh stock buffer at least every six months, or if the pH varies from 7.2 ± 0.2 , or if turbidity indicates microbial growth.

Magnesium Chloride Stock Solution: Dissolve 81.1 g $MgCl_2 \cdot 6H_2O$ with laboratory pure water in a 1 liter volumetric flask. Dilute to a final volume of 1 liter in the volumetric flask with laboratory pure water. Date each batch of stock $MgCl_2$ solution with a date of preparation and store in the refrigerator at 0.0 - 5.0% C for not more than 6 months. Prepare fresh stock solution at least every 6 months or if turbidity indicates microbial growth.



One Set of Records

Some laboratories perform QC tests, record the results on “rough” forms and then transfer the data to the official forms. While this may result in nice, neat records, it is in fact a violation of the rules. Please refer to Chapter 2, Part III of the chemical certification manual. Data should be recorded once-on the official forms only. The problems occur when a laboratory gets behind in transferring the data to the “official” forms. Also if you are ever involved in a court case, your original records will most likely be used. This is also a requirement for microbiological testing laboratories.



Don't Use the Wrong TISAB!

Laboratories certified to perform fluoride testing should not be using concentrated (TISAB III) or powdered TISAB. Use only unconcentrated liquid TISAB for Fluoride tests, mixing the sample and TISAB in equal volumes.



Revisions to Specifications for

Nitrate/Nitrite Determinations

The September 3, 1998 Federal Register contains a drinking water analytical method update which may be accessed at the USEPA website <http://www.epa.gov/fedrgstr>. The revisions in this update become effective on January 4, 1999. The update included the following clarifications regarding the analysis of samples for nitrate or nitrite.



Samples that are chlorinated and/or preserved with sulfuric acid can only be analyzed to determine combined nitrate-nitrite or total nitrate. When present, chlorine and/or sulfuric acid act to oxidize nitrite to nitrate, preventing a separate determination from being made for nitrate or nitrite.

A sample to be analyzed for nitrate or nitrite must be maintained at 4°C or less and have analysis completed within the holding time of 48 hours from the time of sample collection.

A sample to be analyzed for combined nitrate-nitrite must be acidified at the time of sample collection with sulfuric acid and have analysis completed within the holding time of 28 days. If the combined nitrate-nitrite analysis can be completed within 48 hours from the time of sample collection, the sample does not have to be acidified, but it must be maintained at 4°C or less. Acidified samples are not required to be maintained at 4°C or less.

If a laboratory cannot complete analyses for nitrate or combined nitrate-nitrite within 48 hours from the time of sample collection, the samples must be acidified when collected. Laboratories that currently ice, but do not acidify samples must take steps as soon as possible to ensure that this preservation requirement is met. Analytical reports which are not marked Iced, Unpreserved or H₂SO₄ acidified according to this preservation requirement will not be acceptable as of January 4, 1999.

OhioEPA

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