

NC Drinking Water Laboratory Certification Update

2005 Second Release

Certification Update releases

You may have noticed that this is only the second *Certification Update* for the year 2005. The Drinking Water Laboratory Certification Office has experienced turnover in personnel as well as other demands on our time which has limited our ability to prepare this document. Hopefully these newsletters will be released more frequently in 2006.

Personnel & contact changes

As previously mentioned, the Certification Office has had personnel changes in our field auditing staff. Mike King decided to return full-time to laboratory work and in June moved to the Inorganics Laboratory section of the Environmental Sciences Unit. He still remains a key resource on certification issues and also tries to stay current on the certification program. Cindy Price replaces Mike as Supervisor of the Certification Unit. In October Chris Goforth joined the group and is already participating in audits of both microbiological and chemical laboratories. Chris has worked at the State Public Health Lab for over 6 years in the areas of Environmental Microbiology, Lead testing and Bioterrorism Preparedness. Please welcome Chris to our staff when you have the opportunity to do so.

The Certification Office now has a direct phone line and no longer shares a number with the entire Environmental Sciences unit. Our staff can also be reached at our direct numbers; however we suggest that initial contacts be made through the main number where you can be directed to your requested point of contact, or in their absence, your call can be forwarded to another auditor present in the office.

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Reporting

The Certification Office would like to discuss two issues related to reporting. These include the penalty program discussed in detail in previous updates

(attached), and upcoming changes to the reporting formats.

This office continues to work with the Compliance Monitoring Branch of the NC Public Water Supply Section (PWSS) to decrease the number of reports that are late (after the 10th of the month) and those samples with results that are above the allowable limit as stated on the individual reporting forms. Although improved, both of these situations continue to be a problem. In an effort to expedite the review of these reports, PWS is hoping to dedicate personnel to evaluating these forms promptly upon receipt in their office and to make necessary contacts with the Water Treatment Plant (WTP) and/or laboratory before the end of the reporting period. In addition, instead of having a 2 – 3 month grace period before penalties are applied, the Certification Office may allow each laboratory two “free passes”. All correspondence related to laboratory problems will be relayed to the Certification Office through the Head of the Compliance Office.

The North Carolina Public Water Supply Section is in the process of implementing a new database management system known as SDWIS (Safe Drinking Water Information System). This EPA designed program will assist their office in preparing the necessary reports for the EPA in a timely manner. As a result of this new program, the forms used to report data will be changed to include additional required WTP information as well as to amend some of the codes currently used. More details on these required changes will be forwarded in the future. In addition, informational workshops will be planned to discuss these in detail. It is highly recommended that laboratories do not mass produce reporting forms anytime in the near future.

Changes in Certification Requirements

In January 2005 the EPA released the 5th Edition of their *Manual for the Certification of Laboratories Analyzing Drinking Water*. During the past year the Certification Office has been reviewing this manual and noting changes from previous editions. These changes are outlined in the enclosed attachments for both chemical and microbiological testing. Please read these carefully and call or email our office if you have questions. These changes will be incorporated into our checklists in 2006; however, laboratories will be allowed time to make the necessary changes

before deviations are noted. The time allowed will be dependent on the number and difficulty of the changes required. Please note that protocols will be available from our office for selected new quality control tests.

Readycult approval changes

Although mentioned in the attachment, the following changes in Readycult approval are highlighted.

Through the alternate approval process, this test may now be incubated at $35^{\circ}\text{C}\pm 0.5^{\circ}\text{C}$. In addition, use of a 120-mL bottle is allowed. These changes fit more into the routine testing procedures of most analytical laboratories.

Proficiency Evaluation Studies

This is a reminder that a full proficiency evaluation (PE) study must be completed in the first calendar quarter of 2006 unless your laboratory is on an alternate schedule *previously* approved by this office. This study must include all analytes tested by each method for which your laboratory is certified. Each laboratory should refer to their most current status sheet to assure that all the required testing is completed.

If your vendor uses an overnight courier service, please note our street address below:

State Laboratory of Public Health
Certification Office, Room 311
306 N. Wilmington Street
Raleigh, NC 27601

The Certification Office recognizes that some vendor studies will overlap calendar quarters. It is acceptable for a laboratory to start a study in March with the data reported to our office during the second quarter of the year.

Following review of each individual PE study a detailed letter is forwarded to your laboratory contact which outlines (1) what is required in the second quarter as make-up testing for those parameters missed or not reported in the first study; (2) what contaminant/method combinations may have been decertified; and (3) what testing must be completed in the third calendar quarter. Laboratory management is asked to review this information carefully and to call our office if you have questions.

It has been recommended by one of our commercial laboratories that if you are ordering both Water Study (WS) and Water Pollution (WP) ampules, that these be placed on separate orders, even separate phone calls if possible. This laboratory has had numerous problems with data forwarded to the wrong certification office, even when providing detailed information while ordering. This is true for both full studies and make-up sample ampules.

LT2 and D/DBP Rules

The final rule publication for these two programs is expected to be the middle of December, or at the very latest early January. The Certification Office has attempted to keep the laboratory community updated on the testing requirements of the proposed rules, and will continue to provide information when the final rules are released and reviewed, especially when changes have occurred from the proposed to the final document.

2006 Course Presentations

The courses presented by the Certification Office are approved for contact hours for drinking water treatment plant operators. The Certification Office and NCWOA are in the process of submitting these for approval for wastewater contact hours also. The number of hours allowed may vary since parts of these programs specifically addressing drinking water, example reporting, will not be allowed for wastewater. Further information will be available at the time of the course.

Bacteriological Methods for the Examination of Drinking Water:

SLPH, Raleigh: March 7 – 10; June 20 – 23;
November 14 - 17

Process Control Chemistry:

SLPH, Raleigh: February 21 – 24

Lab Tech Day:

McKimmon Center, Raleigh: May 23

Attachments to this Certification Update:

- 1) Certification Changes—Microbiology Section
- 2) Certification Changes—Chemistry Section
- 3) Reprint of Reporting program as outlined in the 2005 Certification update—First quarter

This document has been prepared solely for use by water plants, health departments and commercial laboratories certified by the North Carolina Drinking Water Laboratory Certification Office, State Laboratory of Public Health, to perform compliance testing on public water supply samples.

Reprint from *NC Drinking Water Laboratory Certification Update*—2005 First Quarter

Reporting

The Certification Office has historically relayed reporting questions to the appropriate rule manager in the Compliance Services Branch of the Public Water Supply section and will continue this practice in the future. However, due to increased problems with certified laboratories meeting reporting deadlines (over 800 late bacteriological reports in October) and the continued use of incorrect formats, the Certification Office will now take a more active role in responding to these problems.

Beginning this year, a penalty program will be enforced based on the type of problem and the impact on the affected water treatment plant(s). More specific details will be forthcoming before actual implementation and you are encouraged to read this information thoroughly and call our office if you have questions or concerns.

In general, laboratories that do not report, **within the required timeframe**, any microbial or chemical contaminant above the Allowable Limit or Action Level as defined on the individual PWS report forms, will immediately be dropped to “Provisional” Status. Contaminants considered acute health hazards must be reported the same day as completed, while others which are detected above the stated level must be reported within 48 hours. A second failure to report in a specific time period within one year will lead to “Decertification” of your laboratory for a 30-day period. Before re-certification is awarded, the protocol to be used to prevent a repeat occurrence must be provided to the Certification Office.

Routine reports which are reported late will also be monitored. If a WTP is issued a monitoring and/or reporting violation due to laboratory error, a warning letter will be issued. Failure to again report this type information within a given time period will result in downgrading of the laboratory to “Provisional” status. A third delay, or failure to report as defined in the paragraph above, will result in “Decertification”.

Since reporting problems may overlap multiple contaminant groups, any status changes will cover *all* laboratory testing. Reporting concerns are viewed by the Certification Office in most instances as management problems rather than analytical; therefore any downgrading of certification status will be for the entire laboratory, not just for the contaminant(s) or method(s) that caused the downgrade.

Reporting problems will be tracked over a rolling a 12-month period. Once a laboratory has been decertified for the required 30-day period, the monitoring cycle will start over.

It is not the intent of the Certification Office or the Public Water Supply Section to penalize laboratories due to factors beyond their control. Adjustments in the above plan will be reviewed on a case-by-case basis. The goal of this plan is to prevent undue penalties on the Water Treatment Plants in our state who must publicize violations to the customers served by their facility.

Certification Changes—Microbiology Section

*Manual for the Certification of Laboratories
Analyzing Drinking Water, 5th Edition,*

A number of changes have been made in the Certification Manual since the 4th edition was released in 1997. Major changes which may affect the Total Coliform Rule (TCR) compliance testing performed by laboratories in our state are outlined below. Minor wording changes have not been included. For example, the previous edition often referred to sheen colonies when discussing the membrane filtration procedure; however the new manual refers to “typical colonies” since the newer techniques are not based on sheen production.

The new manual also addresses testing which may be required in the Surface Water Treatment Rule and in the proposed Groundwater Rule. These have not been discussed here since testing under these programs is not currently certified in our state.

The Certification Office will begin implementing these changes immediately; but laboratories will not be determined as deficient until adequate time has been allowed to implement the new requirements. However, if a new method is added by a laboratory, it will be held to the requirements of the 5th edition of the Certification Manual. Please call the Certification Office if you have questions on whether your laboratory will be required to meet any of the new criteria listed below.

Overall comment—anytime a QC record is required, the date and analyst’s initials must be included.

Equipment/Instrumentation

pH Meter (applicable to laboratories preparing media in house)

- The slope of the pH meter must be determined and recorded monthly, after calibration. If the slope is below 95% or above 105%, the electrode or meter may need maintenance. The manufacturer’s instructions should be followed for electrode maintenance and general cleaning. (This QC check has been added to the example pH record form provided by the Certification Office.)

Balance (applicable to laboratories preparing media in house)

- Should provide a sensitivity of at least 0.1 g for a load of 150 g, and 1 mg for a load of 10 g or less.
- The weights used for the monthly calibrations must be re-certified every 5 years with appropriate records and certificates maintained.
- Weights which are damaged or corroded must be replaced (previous manual allowed re-calibration).
- Annual service contracts must include calibration and cleaning in addition to routine maintenance. Provision is made for laboratories which due to geographical location, annual service visits are difficult.

Temperature monitoring devices (applicable to all laboratories)

- Outlines what is required in the calibration records (however these requirements are already part of our program)
- States that the NIST thermometer must be recalibrated every 5 years (previously every 3 years).

Incubator unit (applicable to all laboratories)

- Requires that the time the temperature is read must be included in the QC records.

Autoclave (applicable to laboratories using the autoclave to sterilize media or decontaminate waste materials)

- The records required have been changed to read the time for “an entire cycle (i.e. time between starting autoclave and removing items from autoclave)” in place of requiring the total time for each cycle.
- Requires that a maximum-temperature-registering thermometer be used with each autoclave cycle (previously weekly in the North Carolina program). An electronic readout device is now acceptable.
- Bioindicators (spore strips) must now be used on a monthly basis to confirm sterilization. It will no longer be acceptable to use autoclave tape as the sole indicator of proper autoclave function. Records must be maintained of this testing. The Certification Office can provide information on this quality control procedure upon request (cindy.price@ncmail.net).

Hot air oven (applicable to laboratories using the hot air oven to sterilize glassware)

- Ampoules are no longer recommended for hot air ovens since they may explode or melt. The Certification Office will now require that checks for proper function be performed monthly using spore strips. Records must be maintained of this testing. The Certification Office can provide information on this quality control procedure upon request (cindy.price@ncmail.net).

Refrigerator (applicable to all laboratories)

- Clarifies that the temperature of the refrigerator must be taken on days “the laboratory is staffed”.

Membrane filtration equipment (applicable to laboratories performing the membrane filtration test)

- MF units can be made of porcelain in addition to the previously approved stainless steel, glass or autoclavable plastic.
- Graduation marks must be checked upon initial receipt using a Class B graduated cylinder or better.
- The manufacturer specification sheet for the membrane filters must be maintained and records kept stating that the filters are not brittle or distorted.

Laboratory Practices

Each laboratory should maintain a copy of the MSDS information for every chemical in the laboratory and follow the designated personal protection guidelines for any toxic material.

Sterilization procedures (applicable to laboratories using UV sterilization of MF equipment)

- Ultraviolet light (254 nm) may be used to sanitize equipment between samples. The filtration unit must have been sterilized by autoclaving before initial use.

Sample containers (applicable to all laboratories)

- The sterility test performed on sample containers must be checked for the presence of growth after 24 and after 48 hours incubation. (48 hour check added)

Dilution/rinse water (applicable to laboratories certified for MF and HPC procedures)

- The sterility test performed on each batch of dilution/rinse water must be checked for the presence of growth after 24 and 48 hours incubation. (48 hour check added)

Glassware washing (applicable to laboratories which re-use glassware)

- Each batch of glassware must be checked for pH reactions using 0.04% bromthymol blue indicator. The previous certification manual required that “spot checks” be performed. Records must be maintained.

Analytical methodology

General—Media (parts or entire section applicable to all laboratories)

- If dilution water is used for preparation of serial dilutions, the accuracy of the buffer volume in one dilution bottle/tube in each batch or lot must be checked with appropriate records maintained. The tolerance for a 90-mL or 99-mL bottle is ± 2 mL and ± 0.2 mL for 9-mL tubes.
- The pH of laboratory prepared media must be taken “after sterilization”. The pH of commercially prepared media may be supplied by the manufacturer but must be specific to each individual lot. These records must be maintained in the laboratory quality control files.
- Each new lot of dehydrated or commercially prepared media and each batch of laboratory-prepared medium must be tested before use for sterility and with positive and negative control cultures. In the past the North Carolina program has required that a positive control and a sterility check be performed on each lot or batch, but has not included negative controls (known bacteria cultures that do not give typical or atypical reactions). This will now be required on media used for detection of coliform bacteria. Negative controls are not required for the non-selective media used for bottle sterility checks and for culture maintenance, or for HPC agar.
- Initial testing of each lot of Chromogenic/Fluorogenic reagent material must now include a sterility control.
- Laboratories using commercially prepared media with expiration dates greater than 90 days should run positive and negative controls each quarter, in addition to running these controls along with a sterility control on each lot at the time of receipt.

Multiple tube fermentation (applicable to laboratories certified for the MTF procedure)

- Provides more detail on invalidation of a turbid culture in the absence of gas/acid production. A fecal coliform/*E. coli* positive result is considered a total coliform positive, fecal coliform/*E. coli* positive sample, even if the presumptive or confirmed total coliform test is negative. If the further testing of the turbid sample is negative (total coliform confirmation is negative and fecal coliform/*E. coli* is negative), the sample must be invalidated.

Enzyme (chromogenic/fluorogenic) substrate test (applicable to laboratories certified for a C/F procedure)

- When testing using Colilert, Colilert-18, Colisure, ReadyCult and Colitag, each new lot must be checked for autofluorescence by dissolving a reagent packet in a non-fluorescing vessel and observing using a 365 – 366 nm ultraviolet light.
- The incubation temperature for samples tested using ReadyCult has been changed to $35.0\pm 0.5^{\circ}\text{C}$. In addition, a 120-mL bottle can be used.
- If a large sample load is placed in a small incubator, the recovery time for the unit must be monitored to assure that samples are incubated at 35°C for the total time required. This can be achieved by (1) assuring that samples are at room temperature when placed in the incubator, (2) pre-warming samples to 35°C *before addition of C/F reagent*, or (3) conducting a recovery study to determine the time required for a full load to reach the $35.0\pm 0.5^{\circ}\text{C}$ required incubation temperature and including this time in the laboratory procedure.

Heterotrophic Plate Count (HPC) (applicable to laboratories certified for HPC using pour plate or SimPlate)

- Overall notes now include references to SimPlate
- SimPlate substrate material must have a final pH of 7.2 ± 0.2 .
- Samples tested using SimPlate can be incubated for 45 – 72 hours.
- Pour plate method: Excessive humidity in the incubator must be avoided to prevent the formation of spreaders on the agar surface. At the same time, sufficient humidity must be present to prevent excessive drying of the plates. The laboratory should verify that the water loss during the 48 hour incubation period of HPC plates using the pour plate technique does not exceed 15%. The method to perform this test can be obtained from the Certification Office.

Sample Collection and testing (applicable to all laboratories)

- The 30-hour holding time has been defined as the time between sample collection and placement of samples into the incubator.

Certification Changes—Chemistry Section
Manual for the Certification of Laboratories
Analyzing Drinking Water, 5th Edition,

The Chemistry chapter of the Certification Manual is not as specific as the Microbiology chapter in relation to individual method requirements, and the checklists included in the manual for use by laboratory auditors are general in nature. For our on-site evaluations, the North Carolina Drinking Water Laboratory Certification Office developed forms incorporating the critical components for each method used by laboratories in our state.

The new manual has incorporated the requirements of the Disinfection/Disinfectant Byproducts rule as well as other changes which have occurred since the last publication in 1997 and which have been implemented through the Federal Register and/or 40CFR. These have not been included since the laboratory community has previously been made aware of the new requirements. Also, any change made in the 5th edition which was already part of the North Carolina Certification Program has not been included. Minor wording revisions, unless changing the scope of the statement, may not be referenced.

The Certification Office will begin implementing these changes immediately; but, laboratories will not be determined as deficient until adequate time has been allowed to implement the new requirements. However, if a new method is added by a laboratory, it will be held to the requirements of the 5th edition of the Certification Manual. Please call the Certification Office if you have questions on whether your laboratory will be required to meet any of the new criteria listed below.

Equipment/Instrumentation

Balance

- The weights used for the monthly balance calibrations must meet ASTM Type 1, Class 1 or 2 specifications and should be re-certified every 5 years, or if there is reason to believe that damage (corrosion, nicks) has occurred. Appropriate records must be maintained.
- Each *day* a mechanical or digital balance is used, a verification should be performed consisting of a check using a reference weight at approximately the same nominal weight to be determined.

Temperature monitoring devices

- Liquid-bearing thermometers, such as mercury or alcohol, must be calibrated against a NIST thermometer annually and whenever the thermometer is exposed to temperature extremes. The thermometer must be tagged with the date calibrated and any correction factor (± 0.0 °C if no correction) and records maintained of this testing.
- The NIST thermometer must be recalibrated every 5 years or if subjected to temperature extremes.

- Digital thermometers, thermocouples and other similar electronic temperature measuring devices are to be calibrated at least quarterly. These units should be tagged and records kept as outlined above.
- If an infrared (IR) detection device is used to measure the temperature of the samples, the device should be verified at least every six months using a NIST certified thermometer over the full temperature range that the IR thermometer is used, including at a minimum ambient (20° – 30° C), iced (4 °C) and frozen (-5° – 0 °C). Each day a single check of the IR should be made using a bottle of water, containing a calibrated thermometer, at the temperature of interest. Agreement between the two should be within 0.5 °C or the device recalibrated. Records must be maintained of all testing.

Laboratory Practices

Records maintenance

- Changes in ownership, mergers, or closures of laboratories does not eliminate records maintenance requirements.

Sample collection and shipment

- When temperature is defined in the method as a means of sample preservation, the temperature upon receipt in the laboratory must be taken and documented in the records. Separate temperature control containers, infrared temperature monitoring devices or another means must be determined which will not affect the integrity of the sample. For example, the temperature of the actual sample cannot be taken for those methods such as volatiles organic chemicals (VOCs) and trihalomethanes (THMs) which require zero headspace in the sample container. The procedure(s) to accomplish this requirement must be outlined in a laboratory Standard Operating Procedure (SOP) document.
- When temperature preservation during transport is required, the allowable range is 4° ± 2°C. (Once in the laboratory, these samples must be maintained at 4 °C or less.)

Sample rejection criteria

- The laboratory's criteria for sample rejection must be documented in writing in the laboratory Quality Assurance Plan or in a SOP. The laboratory should reject any sample taken for compliance purposes which does not meet the specifications for
 - Containers and preservatives
 - Holding times
 - Proper collection and/or transport
 - Sample collector
 - Report form (appropriate sections)

The laboratory must notify the facility requesting the analysis and ask for a resample. If re-sampling is not possible and the sample is analyzed, the sample data should be clearly identified in the data packet as being unusable for its intended purpose. In addition, the inadmissibility of these sample data need to be clearly communicated to all end data users.

Analytical methodology

General

- Where safety practices are required in an approved method, they must be followed. The Certification Office may review these during on-site audits.
- Calibration curve—If it is allowable to use a calibration curve for more than one day, (organic methods and some inorganic methods), it must be verified by analysis of at least one standard for each of the target analytes at the expected concentration range. This verification must now be done at the beginning **and at the end** of the analysis run. All checks must be within the control limits required in the method or the system is to be recalibrated and the affected samples reanalyzed. Also, the concentration of the check standard should vary from day to day across the range of analyte concentrations being measured.
- Blanks—**Requirements in the methods must be followed.**
- Laboratory Fortified Blanks (LFB)—**Requirements in the methods must be followed.**
- Laboratory Fortified Sample Matrix (LFM)—**Requirements in the methods must be met.** The North Carolina Certification Rules require analysis of a LFM on a minimum of 10% of the inorganic samples. If there are no LFM requirements in the method, the laboratory should add a known quantity of analytes to a percentage (to be described in the approved SOP) of the routine samples to determine sample matrix interferences.
- The North Carolina Certification Rules currently require that a control sample be analyzed at the **required reporting level** for inorganic methods. The new manual extends this requirement to include all methods, both inorganic and organic. This can be either part of the standard curve or an outside quality control sample. This requirement covers some of the new specifications in the updated manual.
- Initial Demonstration of Capability (IDOC or IDC)—**Requirements in the methods must be followed.**
- Method Detection Limit (MDL) studies—**Requirements in the method must be followed.**

Quality Assurance Plan (QAP)

- In relation to quality control procedures, the laboratory QAP should list the documentation requirements for any required QC procedures specified in the methods for blanks, precision, accuracy, sensitivity, specificity and satisfactory analysis of unknown samples. This can be referenced to the laboratory SOP for each method.
- The QAP must be **read** by all personnel. This must be documented.
- The QAP and/or SOPs need to describe the policies and procedure used by the facility for record integrity in addition to previous requirements for retention and storage.