

3745-89-03

Procedure for laboratory certification.

- (A) General requirements. Laboratories applying for certification to perform analyses to determine compliance with Chapters 3745-81, 3745-82 and 3745-90, and rules 3745-91-06 and 3745-9-09, and as specified by paragraph (B) of rule 3745-83-01 of the Administrative Code, and with plant control tests for one or more public water systems, shall meet the following:
- (1) The laboratory shall submit, for approval by the director, a detailed laboratory plan which includes the following:
 - (a) The analyses for which certification is sought and the number of individuals proposed to perform each analysis.
 - (b) Plans meeting the facility requirements as defined in the "Ohio EPA Laboratory Manual for the Microbiological Analyses of Public Drinking Water ~~2014~~ 2020" and the "Ohio EPA Laboratory Manual for the Chemical Analyses of Public Drinking Water ~~2014~~ 2020."
 - (c) The equipment proposed to be used in the laboratory, including specifications or names, descriptions, manufacturers, and model numbers for each type of equipment.
 - (d) The inventory of standards, reagents and media to be used in analyses for which certification is sought.
 - (2) The laboratory shall submit a quality assurance plan acceptable to the director when certification is sought for drinking water analysis. The "Ohio EPA Laboratory Manual for the Microbiological Analyses of Public Drinking Water ~~2014~~2020" and the "Ohio EPA Laboratory Manual for the Chemical Analyses of Public Drinking Water ~~2014~~ 2020" may be used by laboratories seeking certification for plant control and microbiological testing. Otherwise, an acceptable quality assurance plan shall be developed by the laboratory as described in the United States environmental protection agency's "Manual for the Certification of Laboratories Analyzing Drinking Water" fifth edition, dated January 2005 (designated "EPA 815-R-05-004"), as supplemented in both June 2008 (designated "EPA 815-F-08-006") and November 2012 (designated "EPA 815-F-12-006") by "the United States Environmental Protection Agency, Office of Ground Water and Drinking Water, Cincinnati, OH 45268." The quality assurance plan shall include at least the following parts:
 - (a) Table of laboratory organization which delineates the responsibilities of all laboratory personnel associated with drinking water analyses and

designates the individuals responsible for quality assurance of drinking water analyses in the laboratory.

- (b) Standard operating procedures including identification of the reference methods used to perform the drinking water analyses approved by the United States environmental protection agency.
- (c) Sample handling procedures, including the following:
 - (i) ~~Directions~~ Instructions for maintaining the integrity of the samples by tracking samples from collection to receipt, to and testing to disposal.
 - (ii) ~~Directions~~ Instructions for sample preservation, dechlorination, etc. as required by the reference method and the documentation used by the laboratory to verify that proper sample treatment is done.
 - (iii) ~~Directions~~ Instructions to ensure that adequate sample information is obtained to allow the proper analysis and reporting of results.
 - (iv) Chain of custody forms, where applicable.
 - (v) ~~Directions~~ Instructions for rejecting samples that do not meet shipping, ~~minimum requirements,~~ required reporting information, holding time or preservation requirements and instructions for notifying a public water system which submitted a sample that is rejected for failing to meet minimum requirements.
- (d) Routine practices to maintain the precision and accuracy of data as specified by the director or required by each method of analysis.
- (e) Corrective action procedures taken when unacceptable results are obtained from the analysis of performance evaluation samples or quality control checks.
- (f) ~~Preventative~~ Preventive maintenance procedures including directions and scheduling for instrumentation servicing.
- (g) Documentation of the preparation and expiration of drinking water standards and reagents.
- (h) Reporting procedures including directions followed to ensure that reporting is completed as specified in rule 3745-89-08 of the Administrative Code.

- (3) The laboratory shall submit to the director an application for certification for drinking water analyses required of public water systems, on a form provided by the director. The application shall include the following parts:
- (a) The name, address and telephone number of the laboratory and of the individuals responsible for the laboratory.
 - (b) A list of analyses for which certification is sought. This list shall designate which analytical methods in rule 3745-81-27 or 3745-90-04 of the Administrative Code shall be used for each analysis and shall include the name of each individual who shall perform each analysis.
 - (c) Documentation that the laboratory plan in paragraph (A)(1) of this rule has been accepted by the director.
 - (d) Documentation that the laboratory has obtained acceptable results described in paragraph (B) of this rule ~~and within the acceptance limits described in appendix C to this rule~~ for analyses performed on all appropriate proficiency test samples provided by a proficiency testing provider accredited by a provider of PT samples accredited by a proficiency testing provider accreditor meeting the "National Environmental Laboratory Accreditation Program requirement (NELAP)", and documentation that proficiency test samples were analyzed at least ~~once during each consecutive twelve month period~~ annually for every contaminant by each method for which the laboratory desires certification. The director may designate other acceptable providers of proficiency test samples for analyses to meet this requirement.
 - (e) Payment of the appropriate laboratory survey fee established in accordance with section 3745.11 of the Revised Code. Fees for these surveys shall be paid via a method acceptable to the director. For the purposes of this rule, fees are as follows:
 - (i) The "organic chemicals" fee covers all surveys necessary to obtain laboratory certification for the analysis of drinking water for either of the following:
 - (a) Total trihalomethanes, haloacetic acids, or volatile organic chemicals.
 - (b) Pesticides and other organic chemicals.
 - (ii) The "trace metals" fee covers all surveys necessary to obtain laboratory certification for the analysis of drinking water for all

metals identified by the United States environmental protection agency as primary contaminants, secondary contaminants and contaminants with action levels.

- (iii) The "standard chemistry" fee covers all surveys necessary to obtain laboratory certification for the analysis of drinking water for bromate, chlorite, cyanide, fluoride, nitrate, nitrate-nitrite, nitrite, orthophosphate, total phosphorus, sulfate, total dissolved solids and plant control tests.
 - (iv) The "limited chemistry" fee covers all surveys necessary to obtain laboratory certification for the analysis of drinking water for the following:
 - (a) Any three of the analyses included in standard chemistry.
 - (b) Any two of the analyses included in trace metals.
 - (c) Asbestos.
 - (d) Radioactivity and radioactive chemicals.
 - (e) Beginning one year from the effective date of this rule, total microcystins and cyanobacteria screening.
 - (v) The "microbiological" fee covers all surveys necessary to obtain laboratory certification for the analysis of drinking water for one of the following:
 - (a) MMO-MUG (Colilert or Colisure).
 - (b) Membrane Filtration (MF).
 - (c) Most Probable Number (MPN) by MMO-MUG and MF.
- (4) The director shall return any application which is not filed in accordance with paragraph (A)(3) of this rule.
- (5) Upon the laboratory's successful completion of the requirements of paragraphs (A) (1), (A)(2) and (A)(3) of this rule, the laboratory shall demonstrate acceptable levels of performance during the initial and subsequent on-site surveys, including the following:

- (a) Conformance by the laboratory to the "Ohio EPA Laboratory Manual for the Microbiological Analyses of Public Drinking Water ~~2014~~2020" and the "Ohio EPA Laboratory Manual for the Chemical Analyses of Public Drinking Water ~~2014~~2020."
- (b) Proficiency in appropriate analytical procedures, methodologies, techniques, and use of equipment by analysts participating in the on-site survey.
- (c) Analysis of proficiency test samples.
- (d) Maintenance of laboratory records for at least thirty days prior to the scheduled on-site survey, with the records documenting the following:
 - (i) All appropriate laboratory equipment and auxiliary equipment is operational within prescribed limits.
 - (ii) Sufficient practice analyses have been conducted by each analyst participating in the on-site survey to demonstrate the analyst's proficiency.
 - (iii) An acceptable quality assurance plan has been documented and implemented, as required by paragraph (A)(2) of this rule.
 - (iv) The analyses, quality control procedures, and preparation of standards were correctly performed by each analyst participating in the on-site survey, ~~except for analysts to be designated for operational certification as required for the certification being requested.~~
 - (v) Acceptable method detection limit studies have been completed as required by the director for each method and instrument in accordance with the procedures in ~~appendix A to this rule~~ the 40 Code of Federal Regulations (C.F.R.) Part 136.
- (e) ~~Conformance by the~~ The laboratory conforms to the laboratory plan as approved by the director.
- (f) ~~Conformance by the~~ The laboratory is able to meet the analytical reporting limits listed in tables 1, 2, 3, 4 and 5 in the appendix ~~B~~ to this rule. Laboratories shall report ~~at a~~ to the minimum level that they can consistently quantify but shall not have a minimum reporting limit any higher than the level in the appendix ~~B~~ to this rule. Unless stated in this

rule, the detection limits listed and defined in Chapter 3745-81 of the Administrative Code shall also be used as reporting limits.

(g) Correction of ~~deviations~~violations noted in previous survey reports.

(6) The survey report shall be issued to the applicant by the Ohio environmental protection agency within forty-five days of any on-site survey, shall indicate the acceptability of the applicant's performance during the survey, and shall state ~~deviations~~violations that are required to be corrected prior to certification of the laboratory. If the survey report notes ~~deviations~~violations the director may deny, suspend, or revoke certification in accordance with rule 3745-89-06 of the Administrative Code.

(B) ~~Particular~~ Specific requirements for proficiency testing (PT). Each laboratory Laboratories applying for certification to perform ~~particular~~ drinking water analyses to determine compliance with Chapters 3745-81 and 3745-82, rules 3745-91-06 and 3745-9-09, and as specified by paragraph (B) of rule 3745-83-01 of the Administrative Code, as well as plant control tests shall, in addition to the requirements of paragraph (A) of this rule, include with the application the appropriate required reports as follows:

(1) Applicants for laboratory certification for analytes referenced in paragraphs (C) to (K) of rule 3745-81-27 of the Administrative Code shall report PT results ~~Inorganic chemicals. Applicants for laboratory certification to perform analyses for inorganic chemicals to determine compliance with Chapter 3745-81 of the Administrative Code shall report antimony, arsenic, asbestos, barium, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, thallium, bromate, chlorite, cyanide, fluoride, nitrate, nitrate-nitrite, and nitrite~~ in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results.

(2) Total trihalomethanes. Applicants for laboratory certification to perform analyses for ~~total trihalomethanes~~analytes to determine compliance with rules 3745-81-12 and 3745-81-24 of the Administrative Code shall report bromodichloromethane, bromoform, chloroform and dibromochloromethane analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results. Total trihalomethanes analysis requires reporting all of the compounds established in the definition of total trihalomethanes in rule 3745-81-01 of the Administrative Code.

(3) Volatile organic chemicals. Applicants for laboratory certification ~~to perform analyses for volatile organic chemicals to determine compliance with~~ for analytes referenced in rules 3745-81-12 and 3745-81-24 of the Administrative

Code shall report PT analyses for volatile organic chemicals with maximum contaminant levels (except vinyl chloride) in accordance with paragraph (A)(3)(d) of this rule, with not more than twenty per cent unacceptable results, ~~and shall achieve a method detection limit of 0.0005 milligrams per liter for each of these volatile organic chemicals according to the procedures in appendix A to this rule.~~

~~(4) Pesticides and other organic chemicals. Applicants for laboratory certification to perform analyses for pesticides and other organic chemicals to determine compliance with rules 3745-81-12 and 3745-81-24 of the Administrative Code shall report analysis for pesticides and other organic chemicals in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results.~~

~~(5)~~(4) Microbiological contaminants. Applicants for laboratory certification to perform analyses for ~~microbiological contaminants~~analytes to determine compliance with rules 3745-81-14, 3745-81-21, and 3745-81-41 to 3745-81-45 of the Administrative Code shall report microbiological contaminants analyses in accordance with paragraph (A)(3)(d) of this rule with no more than one unacceptable total coliform result, no more than one unacceptable fecal coliform/E. coli result, and no false negative reported values.

~~(6) Gross alpha particle activity. Applicants for laboratory certification to perform analyses for gross alpha particle activity to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days after receipt, results of the most recent proficiency test for gross alpha particle activity analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results.~~

~~(7) Radium-226 and radium-228 radioactivity. Applicants for laboratory certification to perform analyses for radium-226 and radium-228 radioactivity to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days after receipt, results of the most recent proficiency test for radium-226 and radium-228 analyses in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results, and shall hold laboratory certification under this rule to perform gross alpha particle activity analysis.~~

~~(8) Gross beta particle activity. Applicants for laboratory certification to perform analyses for gross beta particle activity to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days after receipt, results of the most recent proficiency test for gross beta particle activity analyses in accordance with paragraph~~

- ~~(A)(3)(d) of this rule with no unacceptable results, and shall hold laboratory certification under this rule to perform gross alpha particle activity analysis.~~
- ~~(9) Strontium-89 and strontium-90 radioactivity. Applicants for laboratory certification to perform analyses for strontium-89 and strontium-90 radioactivity to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days of receipt, results of the most recent proficiency test for strontium-89 and strontium-90 analyses in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results, and shall hold laboratory certification under this rule to perform gross beta particle activity analyses.~~
- ~~(10) Iodine-131 radioactivity. Applicants for laboratory certification to perform analyses for iodine-131 radioactivity to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days of receipt, results of the most recent proficiency test for iodine-131 radioactivity analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results, and shall hold laboratory certification under this rule to perform gross beta particle activity analysis.~~
- ~~(11) Tritium. Applicants for laboratory certification to perform analyses for tritium to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days of receipt, results of the most recent proficiency test for tritium analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results, and shall hold laboratory certification under this rule for the performance of gross beta particle activity analysis.~~
- ~~(12) Radioactivity from photon emitters (excluding iodine-131). Applicants for laboratory certification to perform analyses for radioactivity from photon emitters (excluding iodine-131) to determine compliance with rules 3745-81-15 and 3745-81-26 of the Administrative Code shall report to the director, within thirty days after receipt, results of the most recent proficiency test for radioactivity from photon emitters (excluding iodine-131) analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results, and shall hold a laboratory certification under this rule to perform gross beta particle activity analysis.~~
- ~~(13)~~(5) Haloacetic acids (five). Applicants for laboratory certification to perform analyses for haloacetic acids (five) analytes to determine compliance with rules 3745-81-12 and 3745-81-24 of the Administrative Code shall report haloacetic acids (five) analysis in accordance with paragraph (A)(3)(d) of this rule with no more than one unacceptable result. Haloacetic acids (five) analysis requires

reporting all of the compounds listed in the definition of haloacetic acids (five) in rule 3745-81-01 of the Administrative Code.

~~(14) Bromate. Applicants for laboratory certification to perform analyses for bromate to determine compliance with paragraph (C) of rule 3745-81-11 and rule 3745-81-23 of the Administrative Code shall report bromate analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results.~~

~~(15) Chlorite. Applicants for laboratory certification to perform analyses for chlorite to determine compliance with paragraph (D) of rule 3745-81-11 and rule 3745-81-23 of the Administrative Code shall report chlorite analysis in accordance with paragraph (A)(3)(d) of this rule with no unacceptable results.~~

~~(16) Plant control tests. Applicants for laboratory certification to perform plant control tests (except chlorine residual) required to be reported by rule 3745-83-01 of the Administrative Code may designate individuals for operational certification. If any individual is designated for operational certification, the application shall also designate an individual responsible for preparation of standards and reagents and for the required monthly and quarterly calibrations and standardizations.~~

(C) The director may issue, deny, suspend or revoke a laboratory certificate in accordance with rule 3745-89-06 of the Administrative Code.

[Comment: This rule incorporates the "Ohio EPA Laboratory Manual for the Microbiological Analyses of Drinking Water ~~2014~~2020" and "Ohio EPA Laboratory Manual for the Chemical Analyses of Public Drinking Water ~~2014~~2020" by reference. Copies are available at www.epa.ohio.gov/ddagw/labcert.aspx and at the "Ohio EPA, Lazarus Government Center, 50 West Town Street, Suite 700, Columbus, OH, 43215." Copies can also be obtained by contacting the laboratory certification office at 614-644-4245.]

[Comment: This rule incorporates the United States environmental protection agency's "Manual for Certification of Laboratories Analyzing Drinking Water" fifth edition, dated January 2005 (designated "EPA 815-R-05-004"), as supplemented in both June 2008 (designated "EPA 815-F-08-006") and November 2012 (designated "EPA 815-F-12-006") by the United States environmental protection agency, office of ground water and drinking water, Cincinnati, OH 45268 by reference. Copies are available at <http://www.epa.gov/dwlabcert> and also at the "Water Resource Center (RC-4100), United States Environmental Protection Agency, 1200 Pennsylvania Avenue NW, Washington, D.C., 20460, (202) 566-1729." Copies can also be obtained by contacting the "Safe Drinking Water Hotline" at 1-800-426-4791. This

document is available for review at "Ohio EPA, Lazarus Government Center, 50 West Town Street, Suite 700, Columbus, OH, 43215."]

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CERTIFIED ELECTRONICALLY

Certification

10/16/2020

Date

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Definition

~~The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.~~

Scope and Application

~~This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.~~

~~The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.~~

~~The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument independent.~~

~~1. Make an estimate of the detection limit using one of the following:~~

~~(a) The concentration value that corresponds to an instrument signal/noise in the range 2.5 to 5.~~

~~(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.~~

~~(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.~~

~~(d) Instrumental limitations.~~

~~It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.~~

~~2. Prepare reagent (blank) water that is as free of the analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.~~

~~3. (a) If the MDL is to be determined in reagent (blank)~~

~~water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.~~

~~(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.~~

~~— If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.~~

~~— If the measured level of analyte is greater than five times the estimated detection limit, there are two options:~~

~~(1) Obtain another sample with a lower level of analyte in the same matrix, if possible.~~

~~(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.~~

~~4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.~~

~~(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:~~

~~(1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.~~

(2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3, and repeat either 4a or 4b.

5. Calculate the variance (S²) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1} \left[\sum_{i=1}^n X_i^2 - \frac{(\sum_{i=1}^n X_i)^2}{n} \right]$$

where:

X_i, i = 1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i = 1 to n.

6. (a) Compute the MDL as follows:-

$$MDL = t(n-1, 1-\alpha=0.99) (S)$$

where:

MDL = the method detection limit

t(n-1, 1-α=0.99) = the student's t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6(a) are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ²/df):

$$LCL = 0.64 MDL$$

$$UCL = 2.20 MDL$$

where:-

LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use S₂ from the current MDL calculation and S₂ from the previous MDL calculation to compute the F ratio. The F ratio is calculated by substituting the larger S₂ into the numerator S_{2A} and the other into the denominator S_{2B}. The computed F ratio is then compared with the F ratio found in the table which is 3.05 as follows: if S_{2A}/S_{2B} < 3.05, then compute the pooled standard deviation by the following equation:

$$6S^2 + 6S^2$$

MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the S_{pooled} as calculated in 7(b) to compute the final MDL according to the following equation:

$$MDL = 2.681 (S_{pooled})$$

where 2.681 is equal to t_(12, 1-α=0.99).

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution:

$$LCL = 0.72 MDL$$

$$UCL = 1.65 MDL$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of replicates (n)	Degrees of freedom (n-1)	t(n-1, 0.99)
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.326

Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water,

do not report a value for the MDL.

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$$S_{\text{pooled}} = \sqrt{\frac{A + B}{12}}$$

Appendix A

[49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986.

If $S_A^2/S_B^2 > 3.05$, respoke at the most recent calculated

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Table 1. Reporting Limits for Analysis of Inorganics	
Analyte	Reporting limit--micrograms/liter (µg/L) except where otherwise noted
antimony	4.0
arsenic	3.0
asbestos	0.2 million fibers/liter (mf/L)
barium	300.0
beryllium	1.0
bromate* ^{4,5}	5.0 or 1.0
cadmium	1.0
chlorine dioxide**	500 50
chlorine (total)**	100
chlorite (Distribution System Monitoring) ion chromatography* ^{1,4}	20
chlorite (Daily Monitoring)* amperometric titration* ²¹	500 100
chromium	10.0
copper	50.0
cyanide	20
fluoride	0.5 milligrams/liter (mg/L)
lead	5.0
mercury	0.5
nickel	20.0
nitrate	0.5 mg/L
nitrite	0.1 mg/L
nitrate-nitrite (as N)	0.5 mg/L
selenium	5.0
thallium	1.5

* disinfection byproduct

** disinfectant residual

Table 2. Reporting Limits for Analysis of Volatile Organic Compounds	
Analyte	Reporting limit--micrograms/liter (µg/L)
benzene	0.5
bromodichloromethane* ^{3,42}	0.5
bromoform* ^{3,42}	0.5
carbon tetrachloride	0.5
chloroform* ^{3,42}	0.5
dibromochloromethane* ^{3,42}	0.5
o-dichlorobenzene	0.5
p-dichlorobenzene	0.5
1,2-dichloroethane	0.5
1,1-dichloroethylene	0.5
cis-1,2-dichloroethylene	0.5
trans-1,2-dichloroethylene	0.5
dichloromethane	0.5
1,2-dichloropropane	0.5
ethylbenzene	0.5
monochlorobenzene	0.5
styrene	0.5
tetrachloroethylene	0.5
toluene	0.5
total trihalomethanes* ^{3,42}	2.0
1,2,4-trichlorobenzene	0.5
1,1,1-trichloroethane	0.5
1,1,2-trichloroethane	0.5
trichloroethylene	0.5
vinyl chloride	0.5
xylene (total)	<u>1.5-5</u>

* disinfection byproduct

Table 3. Reporting Limits for Analysis of Semivolatile Organic Compounds	
Analyte	Reporting limit--micrograms/liter (µg/L)
alachlor	0.2
atrazine	0.3
benzo(a)pyrene	0.10.15
carbofuran	0.9
chlordane - total	0.2
dalapon	5.0
dibromoacetic acid* ^{3,42}	1.0
dibromochloropropane (DBCP)	0.02
dichloroacetic acid* ^{3,42}	1.0
di(2-ethylhexyl)adipate	0.6
di(2-ethylhexyl)phthalate	0.6
2,4-D	1.0
dinoseb	1.0
diquat	2.0
endothall	9.0
endrin	0.1
ethylene dibromide (EDB)	0.01
glyphosate	30.0
haloacetic acids (five)* ^{3,42}	6.0
heptachlor	0.2
heptachlor epoxide	0.1
hexachlorobenzene	0.1
hexachlorocyclopentadiene	0.5
lindane	0.1
methoxychlor	0.1
monobromoacetic acid* ^{3,42}	1.0
monochloroacetic acid* ^{3,42}	2.0
oxamyl (vydate)	2.0
pentachlorophenol	0.4
picloram	1.0
polychlorinated biphenyls (PCBs) - total	0.1
simazine	0.35
2,3,7,8-TCDD (dioxin)	5 x 10 ⁻⁶
toxaphene	1.0
trichloroacetic acid* ^{3,42}	1.0
2,4,5-TP (Silvex)	1.0

* disinfection byproduct

Table 4. Reporting Limits for Radionuclide Analysis	
Analyte	Reporting limit--picocuries/liter (pCi/L) except where otherwise noted
cesium-134	10
gross alpha	3
gross beta	4
iodine-131	1
radium 226	1
radium 228	1
strontium-89	10
strontium-90	2
tritium	1,000
uranium	1 micrograms/liter (µg/L)
other radionuclides	1/10th of the applicable limit

Table 5. Reporting Limits for Analysis of Cyanotoxins	
Analyte	Reporting limit--micrograms/liter (µg/L)
microcystins (total)	0.3

¹ Applicable to monitoring as prescribed in paragraphs (M)(2) to (M)(5) of rule 3745-81-23 of the Administrative Code.

² Reporting limit applicable to operational use only.

²³ When adding individual trihalomethane or haloacetic acid concentrations to calculate total trihalomethane or haloacetic acid, five concentrations, respectively, a zero is used for any analytical result that is less than the minimum reporting limit concentration for the disinfection byproduct.

⁴ The calibration curve must encompass the regulatory minimum reporting level (MRL) concentration. Data may be reported for concentrations lower than the regulatory MRL as long as the precision and accuracy criteria are met by analyzing an MRL check standard at the lowest reporting limit chosen by the laboratory. The laboratory must verify the accuracy of the calibration curve at the MRL concentration by analyzing an MRL check standard with a concentration less than or equal to 110% of the MRL with each batch of samples. The measured concentration for the MRL check standard must be ±50% of the expected value, if any field sample in the batch has a concentration less than 5 times the regulatory MRL. Method requirements to analyze higher concentration check standards and meet tighter acceptance criteria for them must be met in addition to the MRL check standard requirement.

⁵ Laboratories that use EPA Methods 317.0 Revision 2.0, 326.0 or 321.8 must meet a 1.0 µg/L MRL for bromate.

Acceptance Limits for Proficiency Test Samples

Table 1. Acceptance Limits for Inorganic Contaminants

Contaminant	Acceptance limit
Antimony	±30% at ≥ 0.006 mg/L
Arsenic	±30% at ≥ 0.003 mg/L
Asbestos	2 standard deviations based on study statistics
Barium	±15% at ≥ 0.15 mg/L
Beryllium	±15% at ≥ 0.001 mg/L
Cadmium	±20% at ≥ 0.002 mg/L
Chromium	±15% at ≥ 0.01 mg/L
Cyanide	±25% at ≥ 0.1 mg/L
Fluoride	±10% at ≥ 1 to 10 mg/L
Mercury	±30% at ≥ 0.0005 mg/L
Nickel	±15% at ≥ 0.01 mg/L
Nitrate	±10% at ≥ 0.4 mg/L
Nitrite	±15% at ≥ 0.4 mg/L
Selenium	±20% at ≥ 0.01 mg/L
Thallium	±30% at ≥ 0.002 mg/L

Table 2. Acceptance Limits for Disinfection Byproducts

Contaminant	Acceptance limit	Comments
TTHM		
Chloroform-	±20%	Laboratory must meet all 4 individual THM acceptance limits in order to successfully pass a proficiency test for TTHM
Bromodichloromethane	±20%	
Dibromochloromethane-	±20%	
Bromoform	±20%	
HAA5		
Monochloroacetic Acid	±40%	Laboratory must meet the acceptance limits for 4 out of 5 of the HAA5 compounds in order to successfully pass a proficiency test for HAA5
Dichloroacetic Acid	±40%	
Trichloroacetic Acid	±40%	
Monobromoacetic Acid	±40%	
Dibromoacetic Acid	±40%	
Chlorite	±30%	
Bromate	±30%	

Table 3. Acceptance Limits for Lead and Copper

Contaminant	Acceptance limit
Lead	±30% at ≥ 0.005 mg/L
Copper	±10% at ≥ 0.050 mg/L

Table 4. Acceptance Limits for Organic Contaminants

Contaminant	Acceptance limit
DBCP	±40%
EDB	±40%
Alachlor	±45%
Atrazine	±45%
Benzo[a]pyrene	2 standard deviations
Carbofuran	±45%
Chlordane	±45%
Dalapon	2 standard deviations
Di(2-ethylhexyl)adipate	2 standard deviations
Di(2-ethylhexyl)phthalate	2 standard deviations
Dinoseb	2 standard deviations
Diquat	2 standard deviations
Endothall	2 standard deviations
Endrin	±30%
Glyphosate	2 standard deviations
Heptachlor	±45%
Heptachlor epoxide	±45%
Hexachlorobenzene	2 standard deviations
Hexachloro-cyclopentadiene	2 standard deviations
Lindane	±45%
Methoxychlor	±45%
Oxamyl	2 standard deviations
PCBs (as Decachlorobiphenyl)	0-200%
Picloram	2 standard deviations
Simazine	2 standard deviations
Toxaphene	±45%
Aldicarb	2 standard deviations
Aldicarb-sulfoxide	2 standard deviations
Aldicarb-sulfone	2 standard deviations
Pentachlorophenol	±50%
2,3,7,8 TCDD (Dioxin)	2 standard deviations
2,4-D	±50%
2,4,5-TP (Silvex)	±50%