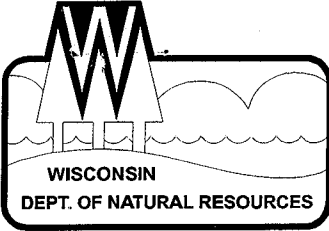


# Clearinghouse Rule 97-141

## State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES



Tommy G. Thompson, Governor  
George E. Meyer, Secretary

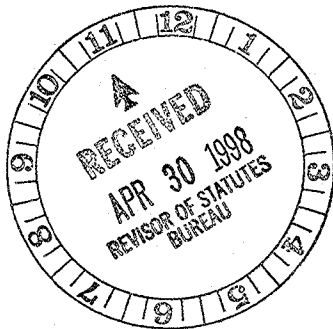
101 S. Webster St.  
Box 7921  
Madison, Wisconsin 53707-7921  
Telephone 608-266-2621  
FAX 608-267-3579  
TDD 608-267-6897

STATE OF WISCONSIN )  
 ) ss  
DEPARTMENT OF NATURAL RESOURCES )

TO ALL TO WHOM THESE PRESENTS SHALL COME, GREETINGS:

I, George E. Meyer, Secretary of the Department of Natural Resources and custodian of the official records of said Department, do hereby certify that the annexed copy of Natural Resources Board Order No. SS-33-97 was duly approved and adopted by this Department on February 25, 1998. I further certify that said copy has been compared by me with the original on file in this Department and that the same is a true copy thereof, and of the whole of such original.

IN TESTIMONY WHEREOF, I have hereunto set my hand and affixed the official seal of the Department at the Natural Resources Building in the City of Madison, this 24<sup>th</sup> day of April, 1998.



George E. Meyer  
George E. Meyer, Secretary

(SEAL)

97-141

Quality Natural Resources Management  
Through Excellent Customer Service



7-1-98



ORDER OF THE STATE OF WISCONSIN NATURAL RESOURCES BOARD TO REPEAL, AMEND,  
REPEAL & RECREATE AND CREATE RULES.

The Wisconsin Natural Resources Board proposes an order to **repeal** s. NR 149.07(6); to **amend** ss. NR 149.02(1), Note and (3), 149.03(5)(c), (d), (l) and (m), 149.03(5) Note, 149.03(25), 149.04 Table 1, 149.05(1)(c), (d) and Table 2, 149.06(1)(b), 149.07(2), 149.07(5), 149.11(3), (5), and (7), 149.14(3)(c)4 and (f)4, 149.21(1)(a), (6)(a), (7)(a) and (8)(a), 149.42(1)(a)6.a., 149.43(3), 149.45(2)(intro.); to **repeal and recreate** ss. NR 149.05(1) Table 2, footnote 1, 149.07(1), 149.07(4), 149.11(1), 149.13; and to **create** ss. NR 149.03(5)(r), (s), (t), (u), (v) and (w), 149.03(8m), Note and (14m), 149.04(1)(a) to (h), 149.04(4), 149.05(5), 149.06(6), 149.09, 149.25, 149.43(4) of the Wisconsin Administrative Code pertaining to laboratory certification and registration.

SS-33-97

Analysis Prepared by the Department of Natural Resources

Statutory Authority: s. 299.11, Stats.

Statutes Interpreted: s. 299.11, Stats.

The effect of amending ch. NR 149, Wis. Adm. Code is to:

1) Specifically require laboratories to be certified or registered with the appropriate agency to submit data for use in a covered program and update the list of covered programs [SECTION 1], 2) Incorporate by reference revisions to the authoritative sources (approved analytical methods) and add new authoritative sources [SECTIONS 2, 3, 4 & 25], 3) Add definitions for the terms "covered program" and "laboratory control sample" and clarify the definition of "reference sample" to be consistent with nationally recognized terms [SECTIONS 5 & 6], 4) Consolidate test category requirements into an easy-to-read list [SECTION 7], 5) Clarify which tests and test categories require reference samples, update test and test category names for consistency, move sulfide from test category 5 to test category 6, add volatile organic compounds and glycols to test category 10, add explosive residues, n-methyl carbamate pesticides, substituted urea pesticides to test category 13, clarify the purpose of test category 19 and create test category 21 for immunoassay certification. Reference samples are not required for platinum, gold, hexavalent chromium and sulfide because they are not readily available from any of the approved reference sample providers. [SECTIONS 8 & 9], 6) Revise the fee formula to allow the program to determine fees up to six months prior to billing, add a fee exemption for laboratories certified only for nitrate and fluoride in safe drinking water, create separate base fees for certification and registration, add fees for category 21 Immunoassay with a relative value of 4, and increase the reciprocity fee to 30 relative value units, [SECTIONS 10 & 11], 7) Allow the Department to adjust application fees to match the actual fees at the time that certification or registration is granted [SECTION 12], 8) Clarify record retention requirements for certified and registered laboratories [SECTION 14], 9) Clarify the procedure laboratories must follow to apply for certification or registration [SECTIONS 15, 16, 17 & 18], 10) Remove the procedure for renewal from the application section and create a new section that clarifies the renewal requirements [SECTIONS 19 & 20], 11) Clarify the requirements for selecting the appropriate analytical methods, sample preservation procedures and holding times [SECTIONS 21 & 22], 12) Clarify and update reference sample requirements, grading criteria and procedures for correcting consecutive reference sample failure [SECTION 23], 13) Specify requirements for approved reference sample providers and remove all requirements for EPA reference samples [SECTIONS 22 & 25], 14) Add quality control requirements for category 21, immunoassay certification or registration [SECTION 26], 15) Update cross references [SECTION

27], 16) Update the code to reflect the transfer of the microbiological certification program to the department of agriculture, trade and consumer protection [SECTION 28], 17) Create a provision allowing for revocation of reciprocity certification in the event the laboratory fails to follow the required quality control procedures [SECTION 29], and 18) Correct miscellaneous technical, spelling and grammatical errors [SECTIONS 13, 21, 24 and 30].

These rules have been submitted to the Department of Agriculture, Trade and Consumer Protection (which replaced the Department of Health and Family Services in the 1995 budget bill for authority to administer the microbiological certification program) and the State Laboratory of Hygiene, as required by s. 299.11(2), Stats. The Department of Agriculture, Trade, and Consumer Protection approved these amendments on January 5, 1998.

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SECTION 1. NR 149.02(1), Note and (3) are amended to read:

NR 149.02(1) This chapter applies to laboratories applying for certification or registration and laboratories holding valid certification or registration, where department rules require laboratory tests to be ~~done~~ performed by a certified or registered laboratory. No laboratory may submit data to the department for use in a covered program unless the laboratory is registered or certified under this chapter to perform the test from which the data was gathered. The department may not accept data from a laboratory that is not certified or registered as required under this chapter except as provided in s. NR 149.44.

**Note:** Administrative rules and programs requiring analyses to be done by a certified or registered laboratory are chs. NR 110- Sewerage Systems, 113- Servicing Septic Systems, 123- Well Compensation Program, 131- Metallic Mineral Prospecting, 132- Metallic Mineral Mining, 140- Groundwater Quality, 145- Private Wells, 150- Environmental Analysis and Review Procedures, 157- Management of PCBs, 158- Hazardous Substance Discharge Notification, 182- Metallic Mining Waste, 206- Land Disposal of Municipal and Domestic Wastewaters, 210- Sewage Treatment Works, 211- General Pretreatment Requirements, 212- Wasteload Allocated Effluent Limits, 214- Land Treatment of Industrial Liquid Wastes, 216- Stormwater Management, 219- Analytical Test Methods, 347- Sediment Sampling and Analysis, ~~508~~507- Environmental Monitoring for Landfills, 605- Identification of Hazardous Waste, 630- Storage, Treatment, and Disposal Facilities, 635- Groundwater Leachate Monitoring, 700 - General Requirements for Investigation and Remediation of Environmental Contamination, 712- Environmental Response Actions, 716- Site Investigation

and, 809- Safe Drinking Water, 811- Design of Community Water Supplies, 845- County Administration of NR 812 (private wells) and HFS 46- Group Day Care Centers for Children.

(3)(a) This chapter does not apply to the certification or registration of laboratories for bacteriological or radiological analyses.

(b) Laboratories shall be certified or approved by the department of ~~health and social services for such testing where department rules require the testing to be done by a certified or approved laboratory~~ agriculture, trade and consumer protection under ch. ATCP 77 to perform bacteriological testing for a covered program.

(c) Laboratories shall be certified or approved by EPA to perform radiological testing for a covered program.

SECTION 2. NR 149.03(5)(c), (d), (l) and (m) are amended to read:

NR 149.03(5)(c) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, November, 1986 (third edition), including ~~December 1987~~, July 1992 (update I), September 1994 (update II), ~~and~~ January 1995 (update IIB), and December 1996 (update III) updates.

(d) "Standard Methods for the Examination of Water and Wastewater", 17<sup>th</sup> 19th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, ~~1989~~ 1995.

(l) "Methods for the Determination of Organic Compounds in Drinking Water", EPA/600/4-88/039, ~~and~~ EPA/600/4-90/020 (supplement I), EPA/600/R-92/129 (supplement II), and EPA/600/R-95/131 (supplement III), Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268.

(m) "Methods for the Determination of Metals in Environmental Samples", EPA/600/4-91/010 and EPA/600/R-94/111 (supplement I), Office of Research and Development, June 1991 and May 1994.

SECTION 3. NR 149.03(5)(r) to (w) are created to read:

NR 149.03(5)(r) "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA/600/R-93/100, EPA, Office of Research and Development, August 1993.

(s) "Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater (Volume I)", EPA 821-R-93-010-A, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, August 1993.

(t) "Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater", EPA/821/R-93-017, Office of Water, Washington, DC 20460, October 1993.

(u) "Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater", EPA/821/B-94-001, Office of Water, Washington, DC 20460, February 1995.

(v) "Method 1631: Mercury in Water by Oxidation, Purge and Traps, and Cold Vapor Atomic Fluorescence Spectrometry" EPA, Office of Water, Washington, DC 20460, January 1996.

(w) "Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels", EPA/821/R-96-011, Office of Water, Washington, DC 20460, July 1996.

SECTION 4. NR 149.03(5) Note is amended to read:

**Note:** Copies of these publications are available for inspection at the offices of the department of natural resources, the secretary of state, and the revisor of statutes. Copies of "authoritative sources" listed in pars. (b), (c), (d), (e), (f), (h), (i), (j), and (k) may be obtained at the addresses given. Copies of "authoritative sources" listed in par. (c) may be obtained from ~~the Government Printing Office, room 190, Federal Building, 517 East Wisconsin Avenue, Milwaukee, Wisconsin 53202~~ US Government Bookstore, 310 West Wisconsin Ave., Suite 150, Milwaukee, WI 53203. Copies of "authoritative sources" listed in pars. (a), (g), (l), (m), (q), (r), (s), (t), (u), (v) and (w) may be obtained from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650. Copies of "authoritative sources" listed in par. (p)

may be obtained from the U.S. Army Environmental Center, Aberdeen Proving Ground, MD, 21020-5401. Copies of "authoritative sources" listed in pars. (n) and (o) may be obtained from the Wisconsin department of natural resources, ~~ERR Section~~ bureau of remediation and redevelopment, 101 S. Webster St., Madison, WI 53707, (608) ~~261-6424~~264-6009.

SECTION 5. NR 149.03(8m), Note and (14m) are created to read:

NR 149.03(8m) "Covered program" has the meaning specified in s. 299.11(1)(d), Stats.

**Note:** Section 299.11(1)(d), Stats., defines "covered program" to include any department program, project, permit, contract or site investigation that requires analytical work be performed by a certified or registered laboratory. See the note in s. NR 149.02(1) for a list of department administrative rules and programs requiring certification under ch. NR 149.

(14m) "Laboratory control sample" means a sample of a known, inert matrix spiked with the analytes of interest.

SECTION 6. NR 149.03(25) is amended to read:

NR 149.03(25) "Reference sample" means a sample used to determine accuracy prepared by a laboratory other than the laboratory conducting the analysis, in which the true value and acceptance limits are unknown to the laboratory at the time of analysis. The term "reference sample" is equivalent to "proficiency testing sample" or "performance evaluation sample".

SECTION 7. NR 149.04(1)(a) to (h) are created to read:

NR 149.04(1)(a) Certification or registration for EP, TCLP or SPLP under test category 7, general III, is for the extraction only and does not include certification or registration for the determinative step. Laboratories shall apply for the determinative steps in the appropriate test categories.

(b) Laboratories shall analyze a representative reference sample for certification or registration under test category 11, organics; semivolatiles by gas chromatography. Representative

reference samples are required for the following subcategories: phenolic compounds (acid-extractables), phthalate esters, nitrosamines, nitroaromatics and isophorone, polynuclear aromatic hydrocarbons, haloethers, nonpurgeable chlorinated hydrocarbons.

(c) Laboratories shall analyze a representative reference sample for certification and registration under test category 12, organics; semivolatiles by gas chromatography/mass spectrometry. To be considered a representative sample, a study shall include at least 4 subcategories of base/neutral extractables in addition to the acid extractables. The base/neutral subcategories include: phthalate esters, nitrosamines, nitroaromatics, isophorone, polynuclear aromatic hydrocarbons, haloethers and nonpurgeable chlorinated hydrocarbons. All semivolatile compounds included in a particular reference sample study shall be analyzed and reported.

(d) Certification or registration for specialized lists of pesticides under test category 14, organics; pesticides is available on a case-by-case basis. These specialized lists include 2,4-D and 2,4,5-TP (silvex) Only and the Resource Conservation and Recovery Act Subtitle D, 40 CFR Part 258, Appendix II (Appendix II of ch. NR 507) organophosphorus pesticide list. The department may require laboratories applying for certification or registration for a specialized list to analyze reference samples for each analyte requested in lieu of analyzing the representative sample for the more inclusive pesticide lists.

(e) Under test category 17, organics; polychlorinated dibenzo-p-dioxin, no reference sample is required for certification or registration. The laboratory shall demonstrate, upon application for certification or registration, acceptable precision and percent recovery for each analyte, based on the successful completion of the initial precision and accuracy portion of the method. The department shall judge acceptability based on the criteria given in the method or the authoritative source.

(f) The list of analytes within test category 18, safe drinking water includes all of the primary and secondary drinking water contaminants having a maximum contaminant level (MCL), specified in ch. NR 809.

(g) Certification or registration under test category 19, any single analyte or group of analytes, is available on a case-by-case basis when the desired certification is not available in the other categories or the analytes are not specifically listed in Table 1. Laboratories shall contact the department prior to applying for certification or registration in this category.

(h) Certification or registration for test category 20, effluent toxicity, includes certification or registration for ammonia, alkalinity and hardness testing as required in the approved procedure. The approved procedure is listed in s. NR 149.22.

SECTION 8. NR 149.04(4) is created to read:

NR 149.04(4) The immunoassay test category has specific requirements which are described in s. NR 149.25.

SECTION 9. NR 149.04 Table 1 is amended to read:

**TABLE 1**  
**Test Categories**

No.	Test Category	Key Analyte - <u>Required Reference Samples</u>	Analytes In Test Category (Includes all forms of the given analytes <u>except where specified.</u> )
1.	Oxygen Utilization	Total BOD <sub>5</sub>	Biochemical <del>oxygen demand</del> <u>Oxygen Demand, carbonaceous biochemical oxygen demand</u> <u>Carbonaceous Biochemical Oxygen Demand.</u>
2.	Nitrogen	Each analyte for which certification or registration is desired except Nitrite.	Nitrate as Nitrogen, Nitrite as Nitrogen, Ammonia as Nitrogen, <del>total</del> Kjeldahl Nitrogen, Nitrate + <u>Nitrate Nitrite as Nitrogen</u>
3.	Phosphorus	Total Phosphorus	Orthophosphate, <u>Total Phosphorus.</u>
4.	Physical	Total Suspended Solids	Total Solids, Dissolved Solids, Volatile Solids, Total Suspended Solids, Oil and Grease.



**TABLE 1**  
**Test Categories**

No.	Test Category	Key Analyte - <u>Required Reference Samples</u>	Analytes In Test Category (Includes all forms of the given analytes <u>except where specified.</u> )
5.	General I	Hardness	Alkalinity/Acidity, Bromide, Chlorophyll a, Color, Hardness, Silica, Silicate, <del>Sulfide</del> , Sulfite, Surfactants.
6.	General II	Each analyte for which certification or registration is desired, <u>except sulfide.</u>	Chemical Oxygen Demand, Chloride, Cyanide, Fluoride, Sulfate, <u>Sulfide</u> , Total Phenolic Compounds.
7.	General III	No reference sample <u>required.</u>	Extraction Procedure (EP) Toxicity, Ignitability, Reactivity, Total Releasable Cyanide, Total Releasable Sulfide, Corrosivity, Waste Fingerprinting Analyses, Total Organic Carbon, Total Organic Halide, Toxicity Characteristic Leaching Procedure (TCLP), Synthetic Precipitation Leaching Procedure (SPLP).  <del>Note: Certification or registration for EP, TCLP, or SPLP under this test category is only for the extraction and does not include the analytes.</del>
8.	Metals I	Each analyte for which certification or registration is desired, <u>except Hexavalent Chromium.</u>	Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, <u>Total Chromium</u> , Cobalt, Copper, <u>Hexavalent Chromium</u> , Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, and Zinc.

**TABLE 1**  
**Test Categories**

No.	Test Category	Key Analyte - <u>Required Reference Samples</u>	Analytes In Test Category (Includes all forms of the given analytes <u>except where specified.</u> )
9.	Metals II	Each analyte for which certification or registration is desired, <u>except Gold and Platinum.</u>	Bismuth, Gold, Iridium, Lithium, Osmium, Palladium, Platinum, Rhodium, Ruthenium, Silicon, Titanium, Tungsten, and Zirconium.
10.	Organics; Purgeable by Gas Chromatography or Gas Chromatography/Mass Spectrometry	Representative purgeable analytes.	<u>Volatile Organic Compounds</u> , Purgeable Halocarbons, Purgeable Aromatics, Acrolein <u>and</u> Acrylonitrile, <u>and</u> Glycols.
11.	Organics; Semivolatiles by Gas Chromatography	Representative analytes within each subcategory for which certification is desired. <del>The following subcategories are included: Phenolic Compounds (acid-extractables), Phthalate Esters, Nitrosamines, Nitroaromatics and Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons.</del>	Phenolic Compounds (acid-extractables), Phthalate Esters, Nitrosamines, Nitroaromatics and Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Nonpurgeable Chlorinated Hydrocarbons.
12.	Organics; Semivolatiles by Gas Chromatography/Mass Spectrometry	Representative <u>semivolatile</u> analytes, <del>within each subcategory for which certification is desired including Phenolic Compounds.</del> (Note: All semivolatiles included in a particular study must be analyzed and reported. To be considered a representative sample for <u>base/neutral extractables</u> , a study must include at least 4 subcategories of <u>base/neutral extractables.</u> )	Phenolic Compounds (acid-extractables) and Base/Neutral Extractable Compounds (excluding pesticides).

**TABLE 1**  
**Test Categories**

No.	Test Category	<u>Key Analyte - Required Reference Samples</u>	<u>Analytes In Test Category (Includes all forms of the given analytes except where specified.)</u>
13.	Organics; Extractables by Liquid Chromatography	Representative Polynuclear Aromatic Hydrocarbons, Benzidines, <u>Explosive Residues and Pesticides analyzable by liquid chromatography.</u>	Acrylamide, Acrylonitrile and Acrolein, Benzidines, Polynuclear Aromatic Hydrocarbons, Aldehydes and Ketones, <u>N-Methyl Carbamate Pesticides subject to Liquid Chromatography</u> (e.g., carbofuran, oxamyl, and methomyl), <u>Substituted Urea Pesticides</u> (e.g., diuron, linuron), <u>and Other Pesticides subject to Liquid Chromatography</u> (e.g., diquat and paraquat), <u>and Explosive Residues</u> (e.g., nitroaromatics, nitroglycerine).
14.	Organics; Pesticides	Representative pesticides within each subcategory for which certification is desired.	Acid Herbicides (e.g., 2,4-D, 2,4,5-T, picloram, etc.), Nitrogen Pesticides, Organophosphorus Pesticides, Triazine Pesticides (including metabolites) and Other Pesticides.
15.	Organics; Petroleum Hydrocarbons	Gasoline Range Organics (GRO), Diesel Range Organics (DRO), Petroleum Volatile Organic Compounds (PVOC).	Gasoline Range Organics, Diesel Range Organics, and Petroleum Volatile Organic Compounds (PVOC).
16.	Organics; Organochlorine Compounds	Representative analytes within <del>the Aroclors and Organochlorine pesticides groups</del> <u>each subcategory</u> for which certification or registration is desired.	Polychlorinated Biphenyls and Organochlorine Pesticides.

**TABLE 1**  
**Test Categories**

No.	Test Category	Key Analyte - Required Reference Samples	Analytes In Test Category (Includes all forms of the given analytes <u>except where specified.</u> )
17.	Organics; Polychlorinated Dibenzo-P-Dioxin	No reference sample, for <del>each analyte for which certification or registration is desired the accuracy and precision data (acceptable according to an authoritative source) shall be submitted to demonstrate the ability to perform the analysis. See s. NR 149.13(11), required.</del>	Polychlorinated Dibenzo-P-Dioxin, Polychlorinated Dibenzo-P-Furan.
18.	Safe Drinking Water	Each analyte or analyte group for which certification is desired.	Cyanide, Fluoride, <u>Sulfide</u> , Metals, Nitrate as Nitrogen, Nitrite as Nitrogen, Nitrate + Nitrite as Nitrogen, Synthetic Organic Contaminants, Total Trihalomethanes, Volatile Organics.
19.	Any Single Analyte or Group of Analytes	<del>That Analyte or Analytes from that Group</del> <u>Each analyte or analyte group for which certification is desired.</u> (where reference samples are available).	Per Request.
20.	Effluent Toxicity	No reference sample <u>required.</u>	Acute Invertebrate Toxicity, Acute Vertebrate Toxicity, Chronic Invertebrate Toxicity, Chronic Vertebrate Toxicity.

**TABLE 1**  
**Test Categories**

No. Test Category	Key Analyte - <u>Required Reference Samples</u>	Analytes In Test Category (Includes all forms of the given analytes <u>except where specified.</u> )
21. Immunoassay	<u>No reference sample required.</u>	<u>2,4-D, 2,4,5-TP, Pentachlorophenol, Polychlorinated Biphenyls, Polynuclear Aromatic Hydrocarbons, RDX, TNT, Toxaphene, Chlordane, DDT, Triazines.</u> <u>(NOTE: Delayed effective date. This category is not available until after September 1, 1999.)</u>

SECTION 10. NR 149.05(1)(c), (d) and Table 2 are amended to read:

NR 149.05(1)(c) The fees for the upcoming fiscal year shall be ~~set~~ based upon program information from the previous fiscal year, and upon the approved spending authority for the upcoming fiscal year. The number of laboratories participating in the program shall be determined no earlier than ~~one month~~ 6 months prior to the billing for the upcoming fiscal year. The estimated travel reimbursement shall be equal to the travel reimbursement from the preceding fiscal year. The calculated fees may not be adjusted during the current fiscal year once laboratories have been billed.

(d) The minimum annual certification fee applies to laboratories certified in any of the test categories 5 through 19, except for laboratories certified only for any combination of nitrate, nitrate + nitrite or fluoride in test category 18, for which there is no minimum annual fee. There is no minimum fee for registration. The department may adjust this fee by the procedures given in pars.

(a) to (c).

<b>Table 2</b> <b>Fees for Certification and Registration</b>	
Item	Relative Value <sup>+</sup>

**Table 2  
Fees for Certification and Registration**

Item	Relative Value <sup>†</sup>
1. <u>Certification</u> Base Fee	<u>4015</u>
2. <u>Registration</u> Base Fee	<u>10</u>
<del>23</del> . Cat. 1 - Oxygen Utilization	1
<del>34</del> . Cat. 2 - Nitrogen	1
<del>45</del> . Cat. 3 - Phosphorus	1
<del>56</del> . Cat. 4 - Physical	1
<del>67</del> . Cat. 5 - General I	2
<del>78</del> . Cat. 6 - General II	2
<del>89</del> . Cat. 7 - General III	4
<del>910</del> . Cat. 8 - Metals I	4
<del>1011</del> . Cat. 9 - Metals II	4
<del>1112</del> . Cat. 10 - Purgeable Organics	4
<del>1213</del> . Cat. 11 - Semivolatiles by GC	4
<del>1314</del> . Cat. 12 - Semivolatiles by GC/MS	4
<del>1415</del> . Cat. 13 - Liquid Chromatography	4
<del>1516</del> . Cat. 14 - Pesticides	4
<del>1617</del> . Cat. 15 - Petroleum Hydrocarbons	12
<del>1718</del> . Cat. 16 - Organochlorine Compounds	4
<del>1819</del> . Cat. 17 - Dioxins	12
<del>1920</del> . Cat. 18 - Safe Drinking Water	20
<del>2021</del> . Cat. 18- NO <sub>3</sub> +NO <sub>2</sub> only <sup>1</sup>	2
22. Cat. 18 NO <sub>3</sub> and Fluoride only <sup>1</sup>	<u>4</u>
<del>2123</del> . Cat. 19 - Any Single Analyte	4
<del>2224</del> . Cat. 20 - Effluent Toxicity Testing	26
25. Cat. 21 - Immunoassay	<u>4</u>
<del>2326</del> . Initial Application	6
<del>2427</del> . Revised Application	3
<del>2528</del> . Minimum Annual Certification & Reciprocity <sup>2</sup> Fee	24

Table 2 Fees for Certification and Registration	
Item	Relative Value <sup>1</sup>
<u>29</u> . Reciprocity Fee <sup>2</sup>	<u>30</u>
<del>26</del> <u>30</u> . Late Renewal Fee (assessed 30 days after payment due date)	2
<del>27</del> <u>31</u> . Evaluation of Out-of-State Labs	<del>Additional</del> Travel Costs
<del>28</del> <u>32</u> . Enforcement Follow-up Evaluation	Actual Cost of Evaluation
<del>29</del> <u>33</u> . Discretionary Acceptance	Actual Cost of Determining Data Quality

SECTION 11. NR 149.05(1) Table 2, footnote 1 is repealed and recreated to read:

<sup>1</sup>This fee applies to laboratories certified for either nitrate (NO<sub>3</sub>) or nitrate + nitrite (NO<sub>3</sub>+NO<sub>2</sub>) in drinking water.

SECTION 12. NR 149.05(5) is created to read:

NR 149.05(5) FEE ADJUSTMENT FOR APPLICATIONS. If an application is not completed within a single fiscal year, the department may adjust the base fee and category fees on the application to recover the difference in fees between the year the application was submitted and the year the application was completed. The laboratory shall pay this difference prior to receiving certification or registration.

SECTION 13. NR 149.06(1)(b) is amended to read:

NR 149.06(1)(b) Quality control ~~data~~ data for spikes, replicates, method blanks, blind standards, reference samples, calibration standards and known standards. Quality control results shall be traceable to all of the associated sample results.

SECTION 14. NR 149.06(6) is created to read:

NR 149.06(6) All certified and registered laboratories shall follow the requirements of this section. Failure to follow these requirements may result in enforcement action, as specified in s. NR 149.42.

SECTION 15. NR 149.07(1) is repealed and recreated to read:

NR 149.07(1) APPLICATION. Initial applications may be submitted by laboratories that do not hold a valid certification or registration under this chapter. Revised applications may be submitted by laboratories that hold a valid certification or registration under this chapter for one or more test categories. Applications for reciprocal certification may be submitted by a laboratory certified by another agency that has established a reciprocal agreement with the department under s. NR 149.43. Laboratories shall submit applications for transfers of ownership as required in sub. (7). The department shall accept any application submitted by a laboratory except as provided in sub (3). A laboratory submitting an application under this chapter shall:

(a) Submit a completed an application form to the department. On the application form, the laboratory shall specify:

1. Whether the application is an initial, revised or transfer of ownership application.
2. Whether the laboratory is requesting certification, registration or acceptance under a reciprocity agreement.
3. The test categories and tests for which certification or registration is requested.
4. The analytical methods the laboratory intends to use to test for each analyte within each test category for which certification or registration is requested. A laboratory shall only use methods which meet the requirements of s. NR 149.11.

(b) Submit the appropriate fees as prescribed by s. NR 149.05. The appropriate fees are determined by the type of application and the test categories requested. The 4 situations are:



1. A laboratory submitting an initial application for certification or registration shall submit the initial application fee, the annual base fee and a fee for each test category requested on the application.

2. A laboratory submitting a revised application for certification or registration shall submit the revised application fee and a fee for each test category requested on the application unless, at the time of the application, the laboratory holds a valid certification or registration for that test category. The department may not charge additional category fees to add tests within a category if the laboratory already holds a valid certification or registration for that category.

3. A laboratory submitting an application for reciprocal certification is exempt from paying the category fees. A laboratory submitting an initial application for reciprocity certification shall submit the initial application fee and the reciprocity fee. A laboratory submitting a revised application for reciprocity certification shall submit the revised application fee.

4. A laboratory submitting an application for transfer of ownership shall submit the revised application fee.

(c) Submit current acceptable reference sample results for each test and test category requested on the application where required in s. NR 149.04 Table 1. The study date for the results shall be no older than 6 months prior to the date the application is received by the department. Reference sample results shall meet the requirements specified in s. NR 149.13.

(d) Submit other analyte specific information as required in the method or the department. Other analyte specific information may include detection limit studies, initial demonstrations of laboratory capability and precision and accuracy studies. The department shall specify on the application form which tests require additional information. The department may request, on a case-by-case basis, any additional information necessary to demonstrate a laboratory's compliance with the requirements of this chapter.

(e) Submit a statement of intent to perform work in Wisconsin. Intent may be manifested by any of the following factors:

1. The laboratory is physically located in the state of Wisconsin.

2. The laboratory is affiliated with a plant, office, laboratory or engineering firm physically located in the state of Wisconsin.

3. The laboratory submits a letter from a potential client requesting analytical work under a covered program.

(f) Submit a current laboratory equipment list.

(g) Agree to comply with this chapter by signing the application form.

(h) Agree to allow the department or its representative to inspect the laboratory to determine compliance with this chapter, with prior notice except as provided in s. NR 149.41(1).

**Note:** Copies of the application form, WI DNR Form 4800-2, are available from the Wisconsin department of natural resources, bureau of integrated science services, PO Box 7921, Madison, WI 53707, (608)267-7633, and on the department of natural resources world wide web site at [www.dnr.state.wi.us](http://www.dnr.state.wi.us).

SECTION 16. NR 149.07(2) is amended to read:

NR 149.07(2) EXPIRATION OF APPLICATION. If the laboratory has not submitted all the necessary materials described in sub. ~~(3)(a) to (c)~~ (1)(a) to (h), ~~its application shall expire~~ within one year from the date the application is received by the department, the application shall expire. If the laboratory then wishes to pursue certification or registration, the laboratory shall pay the appropriate fees and submit a new application ~~per this section~~ as set forth in sub. (1).

SECTION 17. NR 149.07(4) is repealed and recreated to read:

NR 149.07(4) ON-SITE EVALUATION. (a) The department or a department representative shall conduct an on-site evaluation in accordance with s. NR 149.41 of laboratories applying for certification or registration under this chapter. The department shall schedule the on-site evaluation of an applicant laboratory within 90 days of receipt of the completed materials specified under sub. (1) unless a later date is mutually agreed upon by the applicant laboratory and the department. The

laboratory shall respond to and correct the deficiencies cited in the evaluation report prior to receiving their certification or registration.

(b) The department may waive the requirement for an on-site evaluation for a laboratory submitting a revised application if the department has determined that the information submitted with the application indicates that the laboratory is capable of meeting the requirements of this chapter.

(c) Laboratories applying for reciprocal certification under s. NR 149.43 are not required to be evaluated by department personnel prior to receiving certification. Laboratories applying for reciprocal certification shall submit the most recent report from an on-site evaluation performed by the host accrediting agency.

SECTION 18. NR 149.07(5) is amended to read:

NR 149.07(5) ISSUANCE OF CERTIFICATION OR REGISTRATION. The department shall issue the requested certification or registration to the applicant within 20 business days of ~~receipt of the completed application. The application is not considered to be complete until all of the following requirements are satisfied:~~ the date that the laboratory successfully completes the on-site evaluation or the date that the department waives the on-site evaluation. The department may not grant certification or registration until all of the following requirements of the application process are met:

(a) ~~Receipt of~~ The department receives the completed application form as described in ~~subs. (1) and (2), sub. (1).~~

(b) ~~Payment of the annual fee,~~ The laboratory pays the appropriate fees.

(c) ~~Successful performance on reference samples, and~~ The laboratory submits acceptable reference sample results for the analytes listed in s. NR 149.04 Table 1 that require reference samples and for which certification or registration is requested.

(d) ~~Successful completion of an on-site evaluation~~ The laboratory successfully completes an on-site evaluation or the department waives the on-site evaluation.

SECTION 19. NR 149.07(6) is repealed.

SECTION 20. NR 149.09 is created to read:

NR 149.09 RENEWAL OF CERTIFICATION, REGISTRATION OR RECIPROCITY

CERTIFICATION. (1) CERTIFICATION, REGISTRATION AND RECIPROCITY PERIOD. The certification and registration period shall commence on September 1 and end on August 31 of the following year. The department shall renew the certification and registration for laboratories meeting the requirements in sub. (2) or (3) prior to September 1 of each year. Prior to September 1, the department shall request in writing that each certified or registered laboratory submit the fee payment for the next year, acceptable reference sample results and changes in analytical methods used.

(2) CERTIFICATION AND REGISTRATION RENEWAL. In order to renew its certification or registration, a laboratory shall:

- (a) Pay the annual renewal fee.
- (b) Submit acceptable reference sample results for the tests and categories specified in s. NR 149.04(1) Table 1 that require reference samples and for which renewal of certification or registration is requested.

(c) Notify the department of changes in analytical methods and personnel affecting the laboratory's certification or registration, including the lab director, quality assurance officer and laboratory contact person.

(3) RECIPROCITY CERTIFICATION RENEWAL. In order to renew its reciprocity certification, a laboratory accepted under a reciprocal agreement shall:

- (a) Pay the annual reciprocity fee.
- (b) Submit a copy of the host state's letter of accreditation valid for the period for which renewal of reciprocal certification is requested.

(c) Notify the department of changes in analytical methods and personnel affecting the laboratory's certification or registration, including the lab director, quality assurance officer and laboratory contact person.

(d) Submit a copy of the most recent report from an on-site evaluation if the host accrediting agency has performed an on-site evaluation in the previous certification period.

(4) EXPIRATION. The certification or registration for laboratories not meeting the criteria under sub. (2) will expire on the expiration date listed on the certificate. The reciprocal certification for laboratories accepted under a reciprocity agreement not meeting the criteria under sub. (3) will expire on expiration date listed on the certificate.

SECTION 21. NR 149.11(1) and Note are repealed and recreated to read:

NR 149.11 METHODS FOR LABORATORY ANALYSIS, SAMPLE COLLECTION, SAMPLE PRESERVATION, AND HOLDING TIME. (1) Certified and registered laboratories shall follow the methods for laboratory analysis, sample collection, sample preservation and holding times specified in this section. A laboratory shall select the analytical method for a specific test that meets the following criteria:

(a) The method is appropriate for the analyte and sample matrix.

(b) The method is the analytical method required by applicable state and federal regulations or permit, or is approved under s. NR 149.12. If the analytical method is not prescribed by state and federal regulations or permit, the method shall be selected from an authoritative source specified by the department in s. NR 149.03(5).

(c) The method enables the laboratory to quantitate at levels required by the department. If the required level cannot be met by the methods available under par. (b), then the method with the lowest limit of detection shall be selected.

(d) When methods are not available that meet the requirements in pars. (a) to (c), the department may allow methods from other sources.

**Note:** Analytical methods required by state rules are listed in chs. NR 219, 252, 507, 605, 675, 700 and 809. Those required by federal regulations are in 40 CFR 136, 141, 268, and 425.03.

SECTION 22. NR 149.11(3), (5), and (7) are amended to read:

(3) ~~Sample~~ Laboratories shall follow the sample preservation procedures and holding times required by state and federal regulations shall be followed. If sample preservation procedures and holding times are not required by state or federal regulations, laboratories shall follow the sample preservation procedures and holding times established in the analytical methodology shall be followed method. If the analytical ~~methodology method~~ does not establish sample preservation procedures or holding times, laboratories shall follow the procedures in the authoritative sources shall be followed specified in s. NR 149.03(5).

**Note:** Sample preservation procedures and holding times are given in 40 CFR 136, ch. NR 219, SW-846 "Test Methods for Evaluating Solid Waste" as cited in s. NR 149.03(5)(c), and may be specified in the analytical methods.

(5) The limit of quantitation and limit of detection shall be determined for each analyte reported by a laboratory in accordance with a method specified by the department. The department may also require that the limit of detection be determined for a specific matrix.

(7) ~~A copy of the methodology used by the laboratory for each analyte analyzed shall be available to the analyst~~ Laboratories shall make copies of the analytical methods, department regulations and department guidance pertaining to environmental sampling and analysis available to the analysts.

SECTION 23. NR 149.13 is repealed and recreated to read:

NR 149.13 REFERENCE SAMPLES. (1) APPROVED PROVIDERS. The department shall publish a list of approved reference sample providers. The department shall seek the advice of the

certification standards review council prior to approving reference sample providers. The department shall consider the following requirements when approving a reference sample provider:

(a) The provider uses techniques for calculating acceptance limits that are at least as stringent as those used by the Wisconsin state laboratory of hygiene and meet the criteria in sub. (2).

(b) The provider does not release the true values or acceptance limits until the results from all of the laboratories participating in the study are graded and the reports are issued.

(c) The provider agrees to submit results to the department in a format specified by the department, including electronic media.

(2) GRADING CRITERIA. A laboratory's results are acceptable if they are within the approved reference sample provider's acceptance limits. Reference sample results are graded according to the following criteria:

(a) Acceptance limits for all analytes except category 18-safe drinking water shall be determined using the appropriate statistical techniques. If an insufficient number of laboratories participates in the study to generate peer-based acceptance limits, results shall be graded using fixed acceptance limits approved by the department.

(b) Acceptance limits for category 18-safe drinking water analytes shall be as specified in s. NR 149.21.

(c) If a reference sample study contains multiple concentration levels for the same analyte, the laboratory shall achieve acceptable results on greater than 50 percent of the levels to achieve acceptable results for that analyte.

(d) Where certification or registration in a test or test category is based on passing a representative reference sample containing more than one analyte, the laboratory shall report acceptable results on at least 80 percent of the analytes to achieve acceptable results for the study.

(3) LABORATORY REQUIREMENTS. (a) Applications. Laboratories applying for certification or registration shall analyze reference samples from an approved reference sample

provider and submit results for each test or test category included on the application form, as specified in s. NR 149.04 Table 1. Reference sample results shall be graded according to the requirements in sub. (2) and shall be no older than 6 months prior to the date the application is received by the department. The department may not grant certification or registration unless the reference sample results meet the criteria specified in sub. (2).

(b) Annual renewal. Prior to September 1 of each year, certified and registered laboratories shall analyze reference samples from an approved reference sample provider and submit results for each test or test category for which the laboratory seeks renewal, as specified in s. NR 149.04 Table 1. Reference sample results shall be graded according to the procedure in sub. (2). If the results of a reference sample do not meet the criteria specified in sub. (2), the department shall require the laboratory to analyze additional reference samples as specified in sub. (4).

#### (4) PROCEDURE FOR CORRECTING UNACCEPTABLE REFERENCE SAMPLE RESULTS.

(a) All test categories, except category 18- safe drinking water tests. If a certified or registered laboratory does not meet the acceptance limits of an approved reference sample provider for a particular analyte, the department may require the laboratory to analyze a second reference sample if the laboratory does not have acceptable results on a previous sample valid for the same fiscal year. If the results of the second reference sample do not meet the acceptance limits, the department may initiate an assessment of the laboratory's quality control records if this action is necessary to validate data generated by the laboratory. After 2 consecutive reference sample failures the laboratory shall, with the department's approval:

1. Submit a corrective actions report and initiate an action plan to correct the problems within 30 days of the date of notification of the second failure. This action plan shall include a timetable for correcting the problems and obtaining a third reference sample.

2. Qualify all test results of the analytes in the test or test categories which the laboratory has failed to meet acceptance limits on 2 consecutive reference samples. Laboratories shall qualify test results by placing a statement in their analytical report stating that the laboratory has failed 2 consecutive reference samples for this analyte or analyte group.



3. Analyze a third reference sample within the time frame approved by the department. If the results of the third reference sample do not meet the acceptance criteria, the department may not renew the laboratory's certification or registration and may suspend the laboratory's certification as specified in s. NR 149.42. The laboratory shall submit a revised application to have the test or test category reinstated. When applying to have a test or test category reinstated after suspension or non-renewal for 3 consecutive reference sample failures, the laboratory shall provide acceptable results on 2 subsequent and consecutive reference sample studies for that test or test category.

(b) Category 18: safe drinking water tests. If a certified laboratory does not meet the acceptance limits specified in s. NR 149.21, the department may require the laboratory to submit a corrective actions report and analyze a second reference sample. If the results of the second sample do not meet the acceptance criteria, the department may not renew the laboratory's certification and may revoke the laboratory's certification as specified in s. NR 149.42.

(5) RECIPROCITY. Laboratories applying for recognition under a reciprocity agreement as specified in s. NR 149.43 are exempt from the requirements of this section.

SECTION 24. NR 149.14(3)(c)4 and (f)4 are amended to read:

NR 149.14 (3)(c)4. For test category 1, laboratories shall analyze a known standard ~~shall be analyzed~~ after the analysis of 20 samples or once a week. Laboratories that analyze fewer than 20 samples per week shall analyze, at a minimum, one known standard per week. The known standard for biochemical oxygen demand shall be glucose/glutamic acid. The limits on this quality control check shall be as established in an authoritative source or those established by the provider, whichever is more stringent.

(f) 4. Samples for analysis by the toxicity characteristic leaching procedure (TCLP), synthetic precipitation leaching procedure (SPLP) or EP toxicity must be spiked after the extraction at the frequency cited in this paragraph.

SECTION 25. NR 149.21(1)(a), (6)(a), (7)(a) and (8)(a) are amended to read:

NR 149.21 (1) (a) The criteria and procedures for safe drinking water certification are those criteria and procedures specified in "Manual for the Certification of Laboratories Analyzing Drinking Water", ~~EPA/570/9-90/008, third edition, EPA, Office of Water, April 1990, including change 1, October 1991 and change 2, September 1992.~~ EPA 815-B-97-001, fourth edition, EPA, Office of Ground Water and Drinking Water, March 1997.

(6)(a) Analyze reference samples for these substances, ~~provided by EPA~~ obtained from an approved reference sample provider, and achieve quantitative results on the analyses that meet the acceptance limits listed in 40 CFR 141.23 (k) (6) (ii) and 141.89 (a) (1) (ii).

(7) (a) Analyze reference samples which include volatile organic compounds ~~provided by EPA~~ obtained from an approved reference sample provider; and

(8) (a) Analyze reference samples ~~provided by EPA~~ obtained from an approved reference sample provider and meet the acceptance limits listed in 40 CFR 141.24(h)(19)(i)(B).

SECTION 26. NR 149.25 is created to read:

NR 149.25 REQUIREMENTS FOR IMMUNOASSAY CERTIFICATION AND REGISTRATION.

This section applies to laboratories certified or registered under test category 21. (1) The required quality assurance criteria and analytical procedures for immunoassay testing are found in SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" as specified in s. NR 149.03(5)(c).

(2) At a minimum, laboratories certified or registered for immunoassay shall analyze one laboratory control sample and one method blank per 20 samples. If fewer than 20 samples are analyzed per day, the laboratory shall analyze one laboratory control sample and one blank per day.

(3) Laboratories shall demonstrate adequate sensitivity with the chosen immunoassay technique that meets the requirements of the covered program for which the data will be used.

(4) Laboratories are exempt from certification or registration for immunoassay under this chapter if any of the following criteria apply:

- (a) Test results will not be used for compliance with a covered program.
- (b) Test results are used only for in-house quality or process control.
- (c) Test results at a spill site covered under chs. NR 700 to 736 will not be used for site investigation under ch. NR 716 or site closure under ch. NR 720.

**Note:** This section does not become effective until September 1, 1999. The department will accept applications for this category but will not grant certification or registration before September 1, 1999 to allow all interested parties sufficient time to apply.

SECTION 27. NR 149.42(1)(a)6.a. is amended to read:

NR 149.42(1)(a)6.a. Failure of 3 consecutive reference samples for the same analyte or analyte group or failure to analyze the reference samples within the time limit specified in s. NR ~~149.13(8) or (9)~~ 149.13(4)(a)3. Suspension shall only be for the analyte, analyte group or test category in which inability to meet acceptance limits on reference samples or failure to analyze reference samples has been demonstrated.

SECTION 28. NR 149.43(3) is amended to read:

NR 149.43(3) (title) AGRICULTURE, TRADE AND CONSUMER PROTECTION AGREEMENT. The department shall recognize the certification of a laboratory by the department of ~~health and social services~~ agriculture, trade and consumer protection under s. ~~143.15~~ 93.12, Stats., and shall accept the results of any test conducted by a laboratory certified to conduct that category of test under that section.

SECTION 29. NR 149.43(4) is created to read:

NR 149.43(4) REVOCATION OF RECIPROCITY CERTIFICATION. The department may revoke the certification of any laboratory accepted under subs. (1) to (3) if:

- (a) The host accrediting entity suspends, revokes or otherwise terminates the laboratory's certification, or

(b) The laboratory fails to comply with the requirements of this chapter.

SECTION 30. NR 149.45(2)(intro.) is amended to read:

NR 149.45(2) REQUEST FOR VARIANCE. (intro.) A request for a variance shall be submitted in writing to the director, ~~office of technical services~~ bureau of integrated science services, department of natural resources, as far in advance as the situation will permit. Each request for a variance shall contain the following:

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The foregoing rules were approved and adopted by the State of Wisconsin, Natural Resources Board on February 25, 1998.

This rule shall take effect on the first day of the month following publication in the Wisconsin administrative register, as provided in s. 227.22(2)(intro.), Stats., except for section NR 149.25, which shall take effect on September 1, 1999.

Dated at Madison, Wisconsin

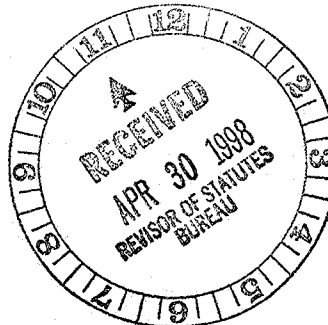
April 24, 1998

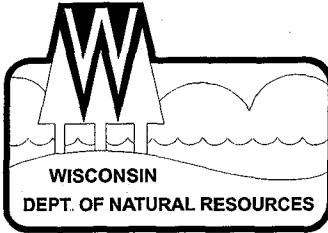
STATE OF WISCONSIN  
DEPARTMENT OF NATURAL RESOURCES

By:

George E. Meyer  
George E. Meyer, Secretary

(SEAL)





State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

Tommy G. Thompson, Governor  
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April 22, 1998

Mr. Gary L. Poulson  
Assistant Revisor of Statutes  
131 West Wilson Street - Suite 800  
Madison, WI

Dear Mr. Poulson:

Enclosed are two copies, including one certified copy, of State of Wisconsin Natural Resources Board Order No. SS-33-97. These rules were reviewed by the Assembly Committee on Natural Resources and the Senate Committee on Agriculture and Environmental Resources pursuant to s. 227.19, Stats. Summaries of the final regulatory flexibility analysis and comments of the legislative review committees are also enclosed.

You will note that this order takes effect following publication. Kindly publish it in the Administrative Code accordingly.

Sincerely,

*George E. Meyer*  
George E. Meyer  
Secretary

Enc.

