

State of North Carolina Department of Natural Resources and Community Development

Division of Environmental Management

February 19, 1987

512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary R. Paul Wilms Director

Ms. Elizabeth Betz, Supervisor Chemist Natural Resources and Environmental Affairs Div. Assistant Chief of Staff Camp Lejeune, NC 28542

Dear Ms. Betz:

We have evaluated results from your analysis of wastewater laboratory certification performance samples received on <u>February 18, 1987</u>. The information from this evaluation is summarized below:

	Value		State		
Parameter	Reported	True Value	Lab Value	Range	Performance
Total Phosphorus	0.12 mg/1	0.118 mg/1			Satisfactory

(X) No additional follow-up is required as all results are acceptable.

() Another sample(s) is included for your analysis.

() You will be provided another sample at a future date.

(X) You will be contacted to establish a date for your on-site laboratory inspection.

() We will issue certification upon receipt of payment for the enclosed invoice.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

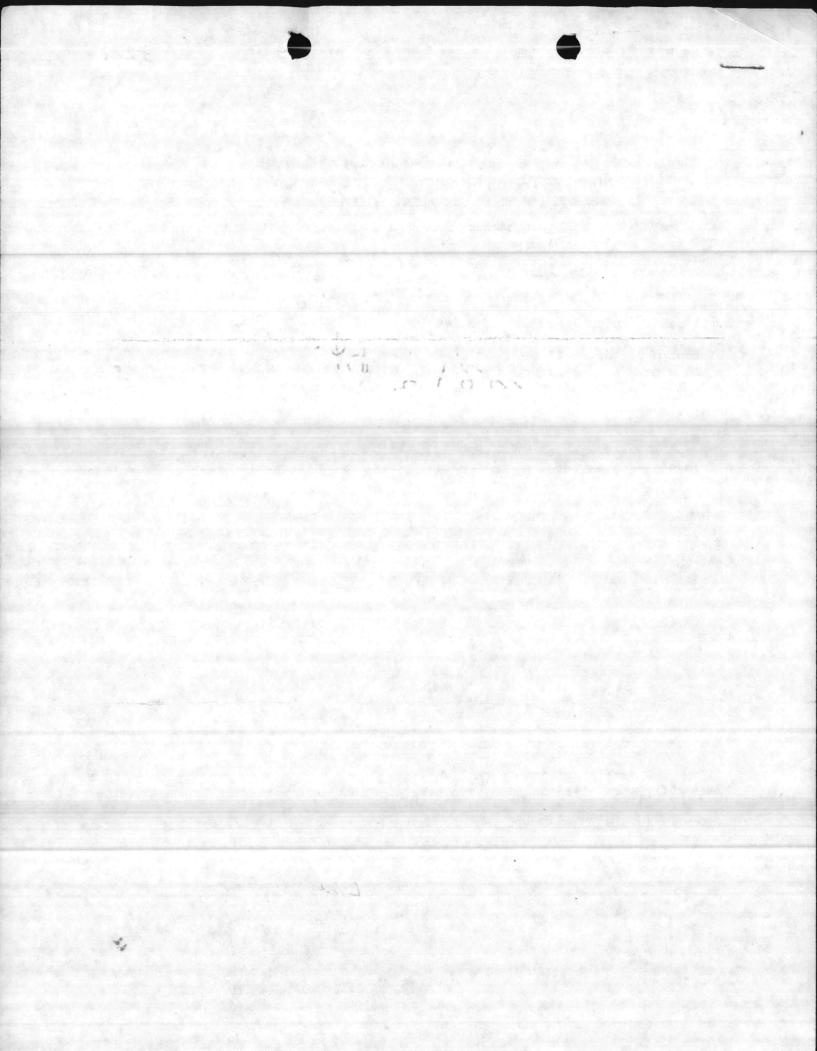
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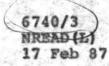
Laboratory Section

cc: Ted Cashion Regional Supervisor, WiRO

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015





Mr. W. B. Edwards NCRD/DEM Laboratory Branch Post Office Box 27687 Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 19 January 1987 letter, the Certified Laboratory Performance Analysis Report is submitted. Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely,

J. I. WOOTEN Director

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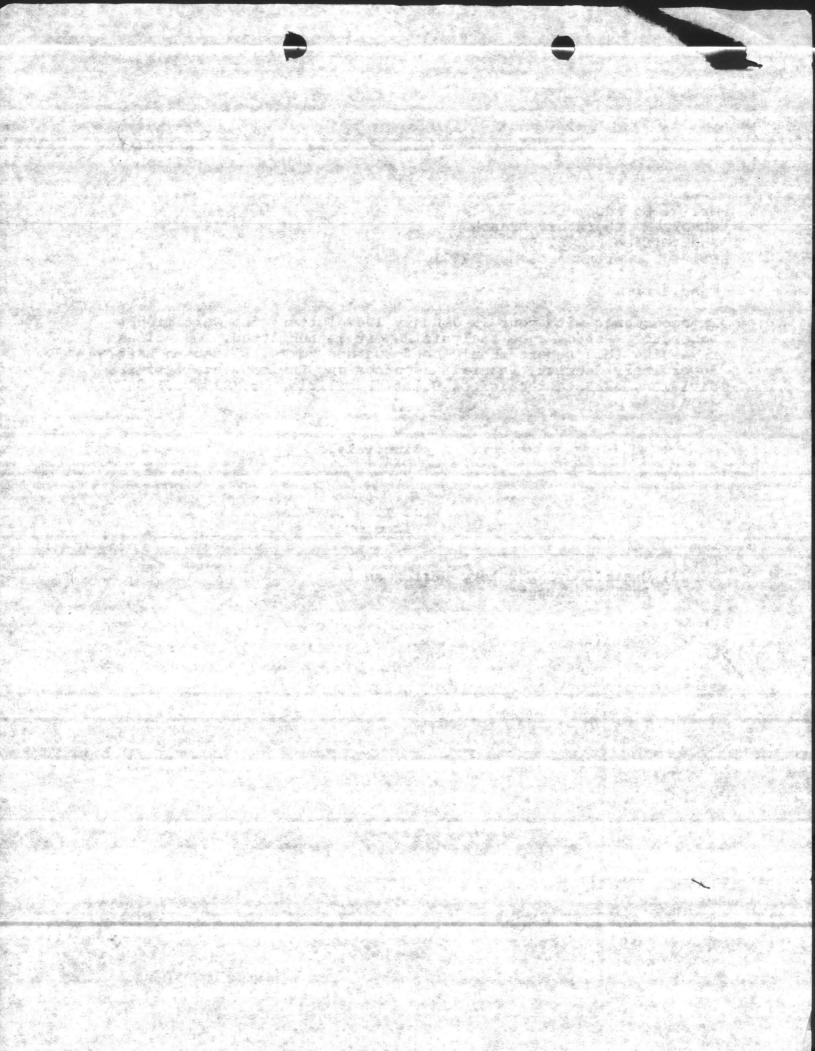
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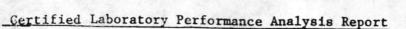
(1) Certified Laboratory Performance Analysis Report

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States and

Copy to: ECML, NREAD (2)





11987

Cert No

Date 2-11-87

This form is to be used to report the results of your analysis of the enclosed performance evaluation samples. One completed copy of this form is the only report needed. Do not return the individual report forms or mailing cartons. In the appropriate space below, enter the dilution made on the ampul contents (example: 5/1000 for 5 ml to 1000 ml), the value obtained from your analysis, and return this report to:

> NRCD/DEM Laboratory Section P. O. Box 27687 Raleigh, MC 27611-7537

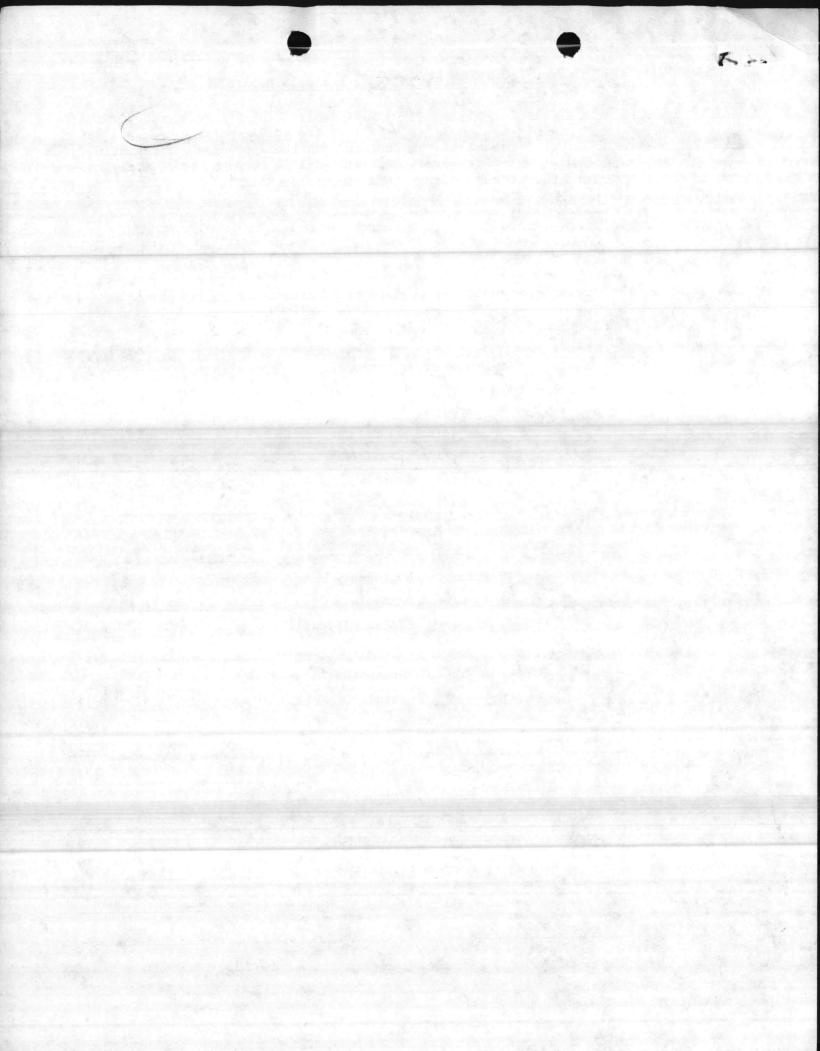
within 30 days of your receiving date. Reports received after this 30 day period will be considered unsatisfactory.

Parameter	Dilution Made	Value Obtained	Parameter	Dilution Made	Value Obtained
BOD		mg/1	Metals, Group II:		
COD		mg/1	Antimony		µg/1
Chloride		mg/1	Silver		μg/1
Cyanide		mg/1	Thallium		μg/1
Fluoride		mg/1	Arsenic		μg/1
Grease & Oil		mg/1	Barium		µg/1
Hardness		mg/1	Mercury		μg/1
MBAS		mg/1	Selenium		µg/1
Metals, Group I:			Nitrogen:	Second Park	PB/ 1
Aluminum	19 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	μg/1	Ammonia		mg/1
Beryllium	THE ST.	µg/1	Total Kjeldahl		mg/1
Cadmium	he for the	μg/1	Nitrate + Nitrite	,	mg/1
Chromium		μg/1	Phosphorus:		mg/1
Cobalt		μg/1	Total (as P))	10/1000	- 12 11
Copper		μg/1	Ortho (as P)	10/1000	<u>0,12 mg/1</u>
Iron	Caller -	μg/1	pH		mg/1
- Lead	Calling and	μg/1	Phenols		Units
Manganese		μg/1	Residue:		µg/1
Nickel		We will also a strategy and the strategy and			and the second
Zínc		µg/1	Total	<u> </u>	mg/1
-	and the second s	µg/1	Suspended		mg/1
			Turbidity		NTU

Laboratory reporting data FNVIRONMENTIL CHEMISTRY MICROBIOLOGY LABORATORY

13

Signature 210, 210760 Bt. LABORATORY SUPERVISOR





State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management

Division of Environmental Ivianagement

512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary

January 19, 1987

R. Paul Wilms Director

Ms. Elizabeth A. Betz Director, Natural Resources Div. Environmental Chemistry & Microbiology Lab Camp Lejeune, NC 28542

Dear Ms. Betz:

We have evaluated your results from the performance samples for wastewater laboratory certification received on <u>January 9, 1987</u>. The information from this evaluation is summarized below:

Parameter	Value Reported	True Value	State Lab Value	Acceptable Range	Performance
*Total Phosphorus	7.22 mg/1	3.37			Unacceptable
Ammonia Nitrogen	8.23 mg/1	8.56			Acceptable

() Your laboratory will be recommended for (certification) (recertification).

() You will be contacted to establish a date for your on-site laboratory inspection.

(*) Another sample(s) has been included for your use as required.

() You will be provided another sample at a future date.

Contact us at 919-733-3908 if you have questions concerning this matter.

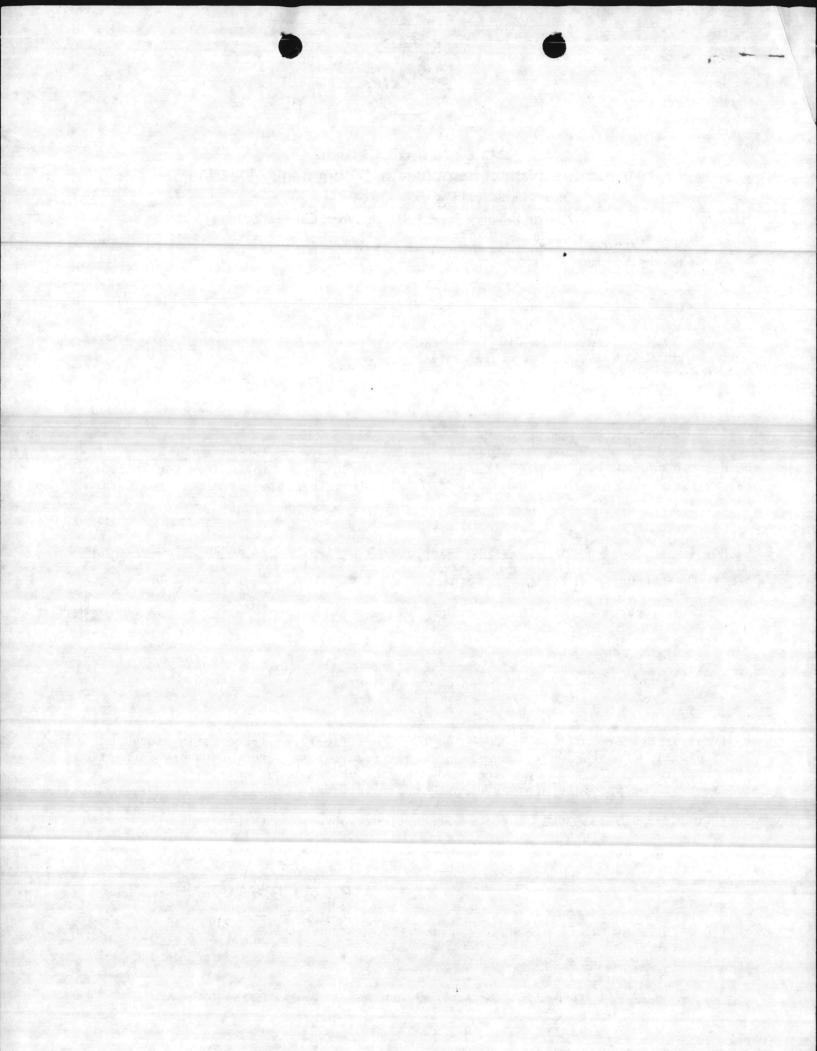
Sincerely,

W. B. Echvarol

Laboratory Section

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



6740/3 NREAD 8 Jan 87

Mr. W. B. Edwards NCRD/DEM Laboratory Branch Post Office Box 27687 Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 9 December 1986 letter, the instruction and reporting form for nitrogen and phosphorus analysis is submitted. The evaluation samples were received on 15 December 1986.

Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

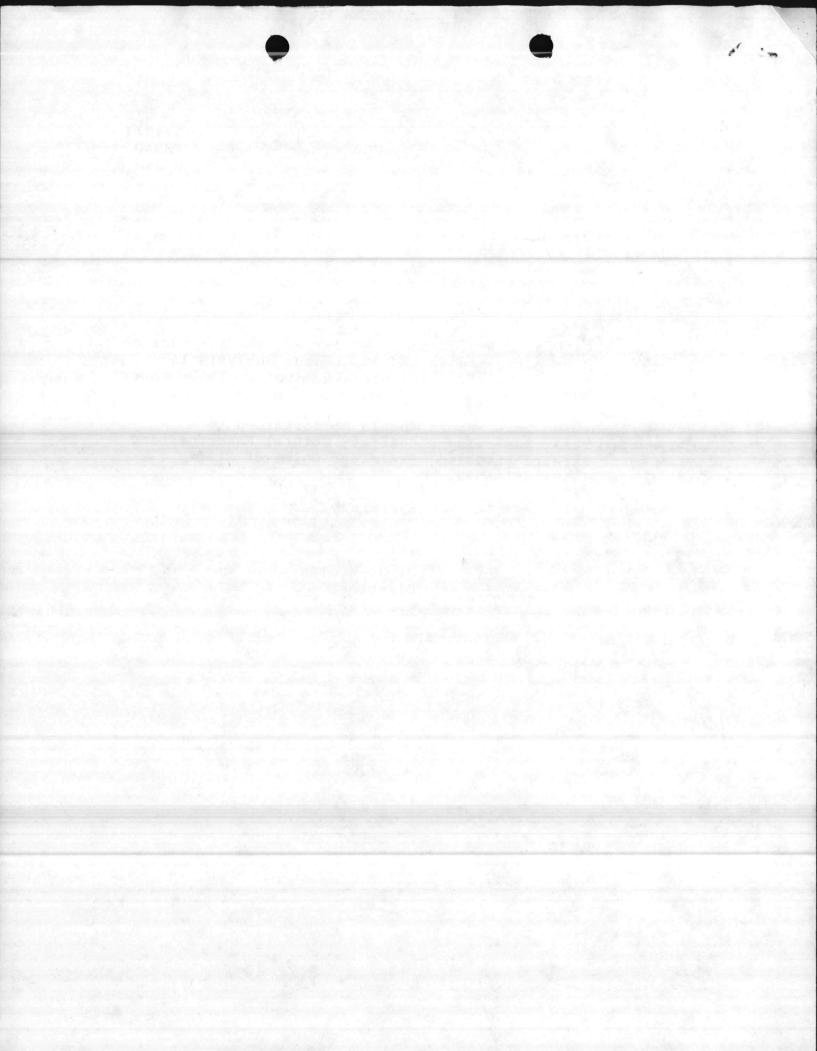
Sincerely,

J. I. WOOTEN Director

Encl:

(1) Instruction & Repting Form for Nitrogen and Phosphorus Analysis

Copy to: ECML, NREAD (2)



Instructions and Reporting Form for Nitrogen and Phosphorus Analysis

The contents of the enclosed ampul(s) are to be used to prepare analytical performance samples for Total Kjeldahl nitrogen and total phosphorus or ammonia nitrogen, nitrate + nitrite nitrogen, and ortho phosphorus analyses. When you are prepared to perform the analyses, open the ampul and transfer 10.0 ml from it to a one liter volumetric flask. Add distilled or deionized water to volume and mix thoroughly. See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Please verify that the ampul you received is NUTRIENT 4.

Perform Analyses Marked (X)	C. Analysis	Range mg/1	Report Results mg/1	Value Obtained mg/1
•	Total Kjeldahl Nitroge	n 0.1 - 10	to 0.01	
X	Total Phosphorus	0.05 - 10	to 0.01	7.22

Please verify that the ampul you received is NUTRIENT 2.

Perform Analyses Marked (X)		ntration ge mg/1	Report Results mg/1	Value Obtained mg/1
	Nitrate + Nitrite Nitroger	0.05-10	to 0.01	
×	Ammonia Nitrogen	0.05-10	to 0.01	8.23
	Ortho Phosphorus	0.05-10	to 0.01	

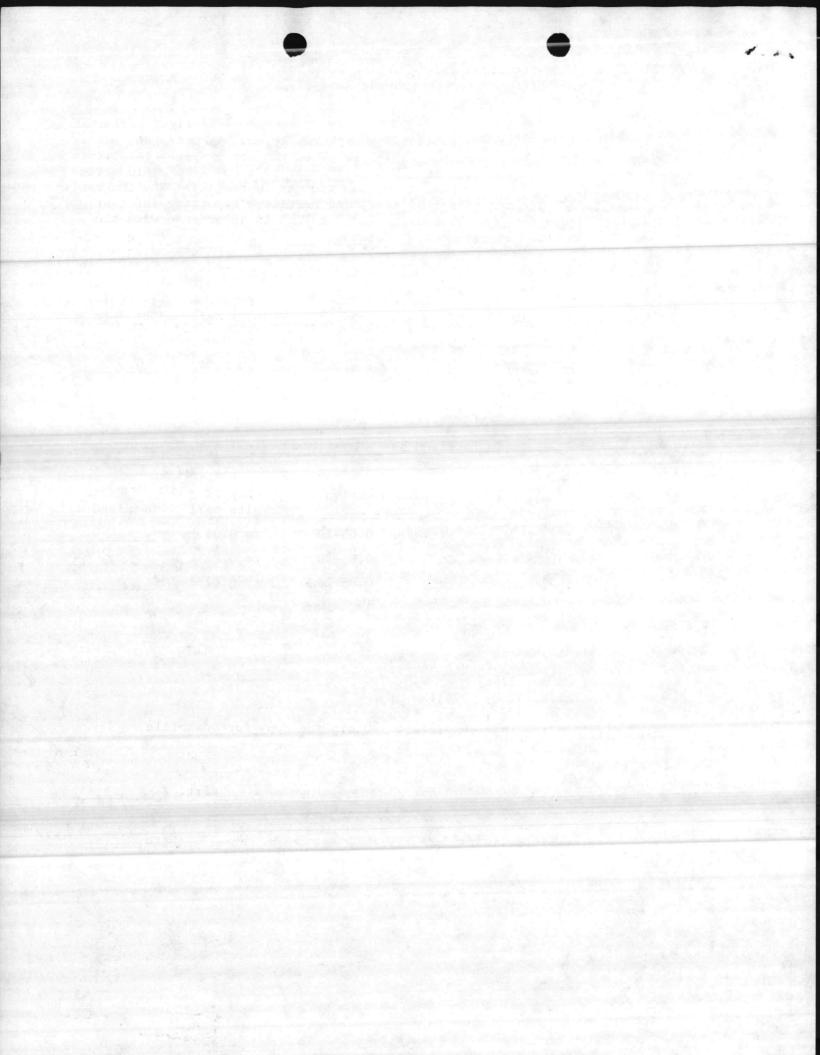
Return a signed copy of this completed form to:

W. B. Edwards NRCD/DEM Laboratory Section P. O. Box 27687 Raleigh, N. C. 27611

within 30 days of receiving this sample(s). Retain one copy for your file.

Laboratory reporting of	data ENVILONMENTAL	CHEMISTRY +	Cert. #	
	MICROBIDLOGY			
Signature of Laborato	ry Supervisor Ma	abeth a B	Date 22	Dec 1986

(LC-31-Rev.86)





State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management

512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary January 7, 1987

R. Paul Wilms Director

Ms. Elizabeth A. Betz Director, Natural Resources Div. Environmental Chemistry & Microbiology Laboratory Camp Lejeune, NC 28542

Dear Ms. Betz:

We have evaluated results from your analysis of performance samples received on October 29, 1986 . The information from this evaluation is summarized below:

Parameter	Value Reported	True Value	State Lab Value	Acceptable Range	Performance
Oil & Grease	37.0 mg/1	40 mg/1			Satisfactory
Suspended Residue	21.5 mg/1	21 mg/1			"

(X) All results are acceptable, therefore, no additional follow-up is required.

() Upon receipt of payment for the enclosed invoice, we will issue certification.

() Your laboratory will be recommended for certification.

() Another sample(s) has been included for your analysis as required.

() You will be provided another sample at a future date.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

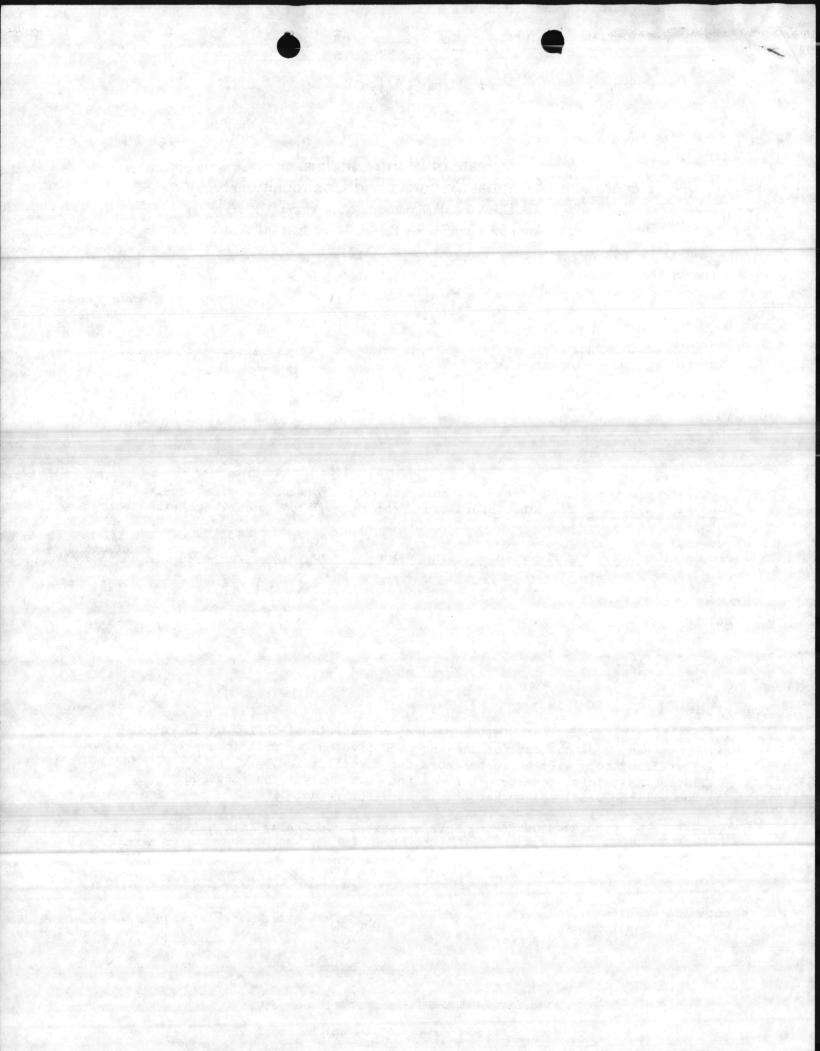
Billy D. Bynd

Laboratory Section

cc: Ted Cashion WiRO Supervisor

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



NH3 + Total P sample enclosed per telephone discussion with Mrs. Betz UW & W. B. Edwards

December 9, 1986

Mr. Julian I. Wooten Director, Natural Resources Div Environmental Chemistry & Microbiology Laboratory Camp Lejeune, NC 28542

Dear Mr. Wooten:

North Carolina Wastewater Laboratory Certification Re:

We have received and reviewed your December 4, 1986, letter concerning laboratory certification. We have amended our files to reflect your current laboratory name and staff listing supplied with your letter. Attached is a report for the evaluation sample analysis performed by your laboratory. As shown on the report, we have enclosed Grease & Oil and total suspended residue rerun samples for your analysis. Also enclosed are initial evaluation samples for total phosphorus and ammonia nitrogen analysis. Acceptable results are required on all parameters prior to certification.

Thank you for the information supplied. Contact us at 919-733-3908 if you have questions.

Sincerely,

WBE

W. B. Edwards Laboratory Section

From

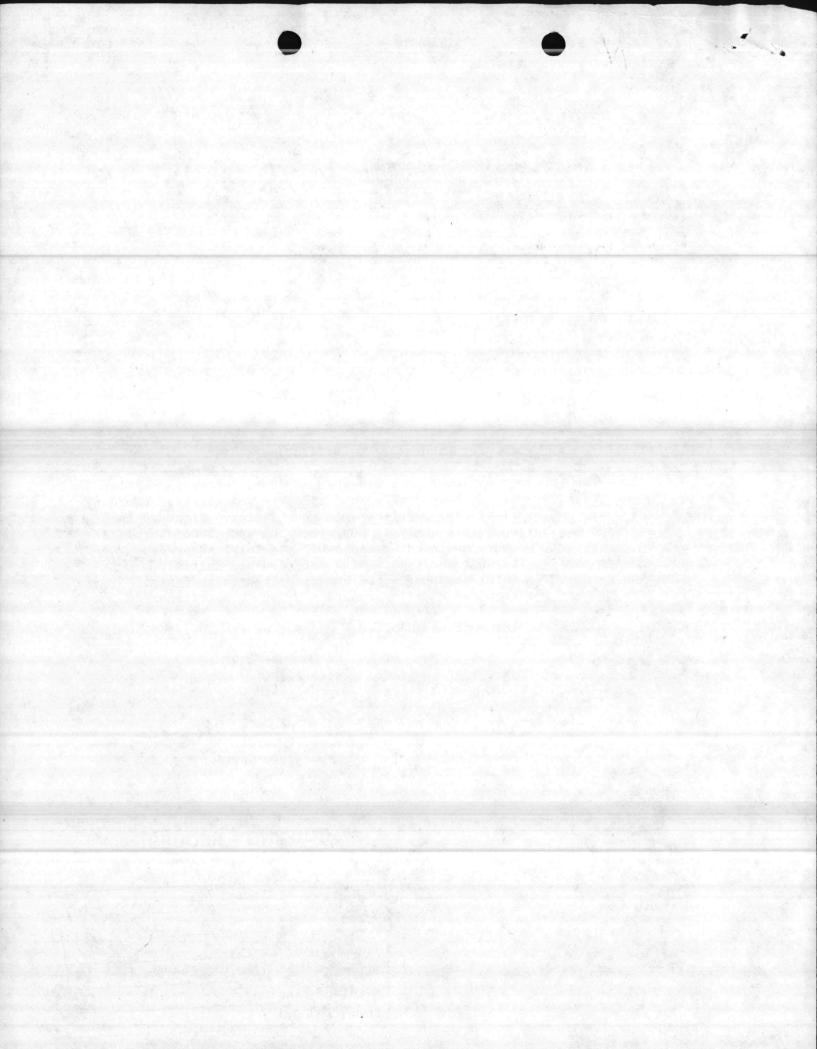
Enclosures

cc: Wilmington Regional Supervisor Billy Byrd Please Scotch® 7664 "Post-it" Routing-Request Pad

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Instructions and Reporting Form for Nitrogen and Phosphorus Analysis

The contents of the enclosed ampul(s) are to be used to prepare analytical performance samples for Total Kjeldahl nitrogen and total phosphorus or ammonia nitrogen, nitrate + nitrite nitrogen, and ortho phosphorus analyses. When you are prepared to perform the analyses, open the ampul and transfer 10.0 ml from it to a one liter volumetric flask. Add distilled or deionized water to volume and mix thoroughly. See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Please verify that the ampul you received is NUTRIENT 4

Perform Analyses Marked (X)	Analysis	Concentration Range mg/1	Report Value Results mg/1 Obtained mg/1
·	Total Kjeldahl Nitrog	en 0.1 - 10	to 0.01
<u> </u>	Total Phosphorus	0.05 - 10	to 0.01 7.22

Please verify that the ampul you received is NUTRIENT 2

ceived and reviewed your Wederstor 4. 1986, let lication. We have amended our files to reflect your en T. Teller Perform Analyses Sand actant in Concentration were in Report trached Value Marked (X) Analysis Range mg/1 Results mg/1 Obtained mg/1 Nitrate + Nitrite Nitrogen 0.05-10 to 0.01 STARFE Ammonia Nitrogen 0.05-10 to 0.01 0.05-10° prior to 0.01 Ortho Phosphorus schon.

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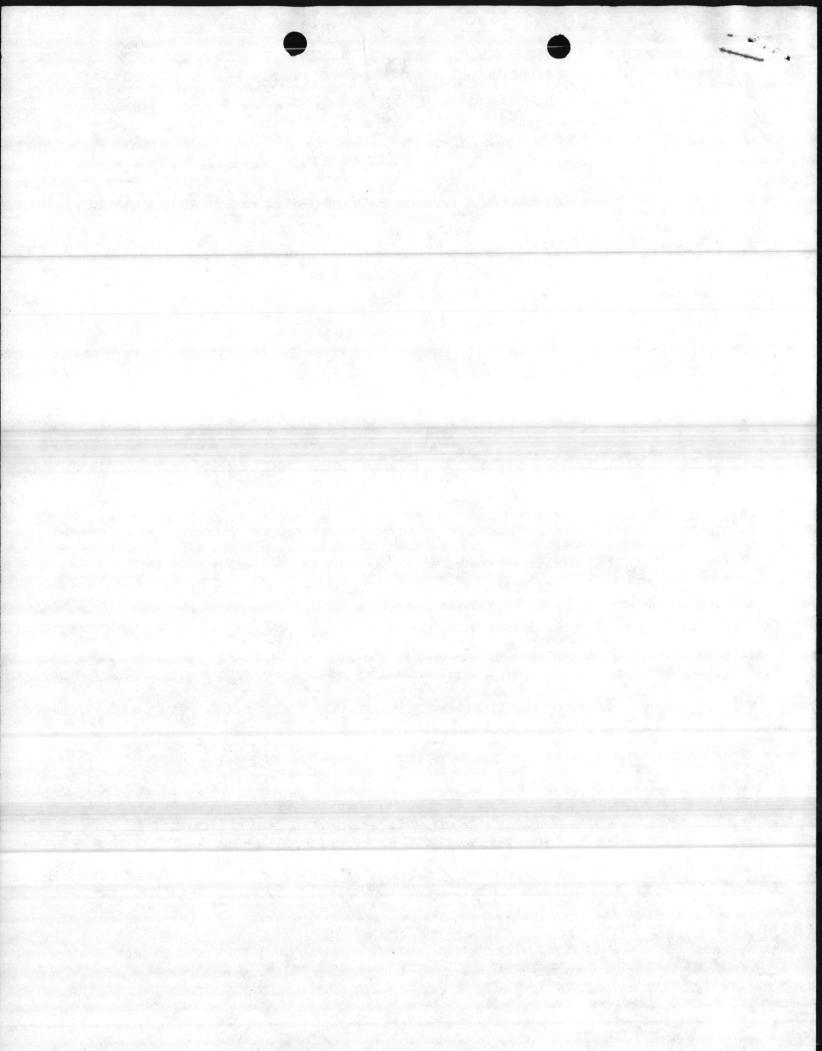
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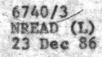
Return a signed copy of this completed form to: Context us at 910-738-3908 if

W. B. Edwards NRCD/DEM Laboratory Section P. O. Box 27687 Raleigh, N. C. 27611

within 30 days of receiving this sample(s). Retain one copy for your file.

Laboratory reporting data Enviro	NMENTAL CHEMISTRY &	Cert. #
Mick	BIDLOGY LABORATORY	
Signature of Laboratory Superviso	- Elizabeth OBit	Date 22 Dec 1986
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Mr. W. B. Edwards NCRD/DEM Laboratory Branch Post Office Box 27687 Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 9 December 1986 letter, the instruction and reporting forms for analysis of suspended residue and oil and grease are submitted.

The initial evaluation sampled for total phosphorus and ammonia nitrogen were received on 15 December 1986. Their results will follow later.

Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely,

D. D. SHARPE Acting Director

Encls:

- (1) Instruction & Rpting Form for
- Analysis for Suspended Residue (2) Instruction & Rpting Form for Analysis of Oil & Grease

Copy to: ECML, NREA



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Instruction and Reporting Form for Analysis of Total and Suspended Residue

Laboratory	Code #	Certificate #	
Laboratory	reporting data Environm	MENTAL CHEMISTRY AND MICEOBISLOCY LABO	SCATOLY
Signature o	of Laboratory Supervisor_	Elizabeth aby	
Date Comple	eted 15 December 1986		

See the table below for information on the concentration range and pertinent reporting

NOTE: Some labs have found it necessary to correct for their distilled water residue

information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Perform analyses Marked (X)	Analysis	Concentration • Range	Report Results	Value Obtained
	Total Residue	1-1000 mg/1	to 1.0 mg/1	
X	Suspended Residue	1-1000 mg/1	to 1.0 mg/1	215

the sample up to the 1000 ml mark.

Mix well and analyze by standard procedures.

content when analyzing this type of sample.

Dept. of Natural Resources & Community Development Laboratory Section Division of Environmental Management P. O. Box 27687 Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Return a signed copy of this completed form to:

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rough a glass funnel into the 1000 ml volumetric flask. ively rinse the solids from the vial and rubber seal e the glass funnel by adding deionized water to make k.

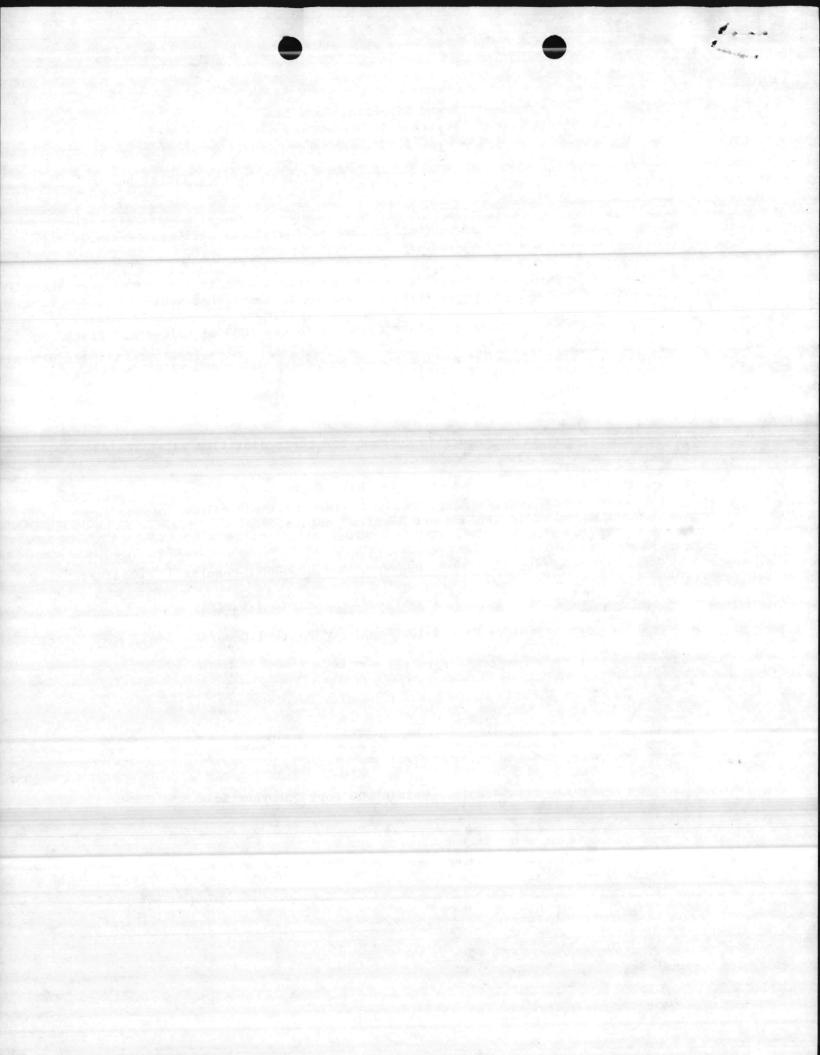
Please verify that the ampul you received corresponds with the number listed below: R

for total and suspended residue analysis.

When you are ready to perform the analysis, remove the rubber seal from the vial containing the solids, being careful not to lose particles clinging to the r

The contents of this ampul are to be used to prepare an analytical performance sample

(LC7-4/81)



Instruction and Reporting Form for Analysis of Oil and Grease

Please verify that the number on the ampul you received corresponds with the number listed here:

The contents of this ampul are to be used to prepare a sample for the analytical performance test for oil and grease analysis. Add 500 ml of distilled or defonized water and 2.5 ml concentrated HCl to a separatory funnel. Transfer/0.0 ml of the ampul contents to the separatory funnel. Complete the analysis as detailed in the required procedure. Calculate the oil or grease concentration using the formula below: 20.5 1.3 mg/l grease or oil = (mg oil or grease in sample - mg oil or grease in blank) x 1000

500

Please record your result in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Perform analyses Marked (X)	Analysis	Concentration R	Report Results	Value Obtained		
	Oil & Grease	1-1000 mg/1	to 1 mg/1	37.0		

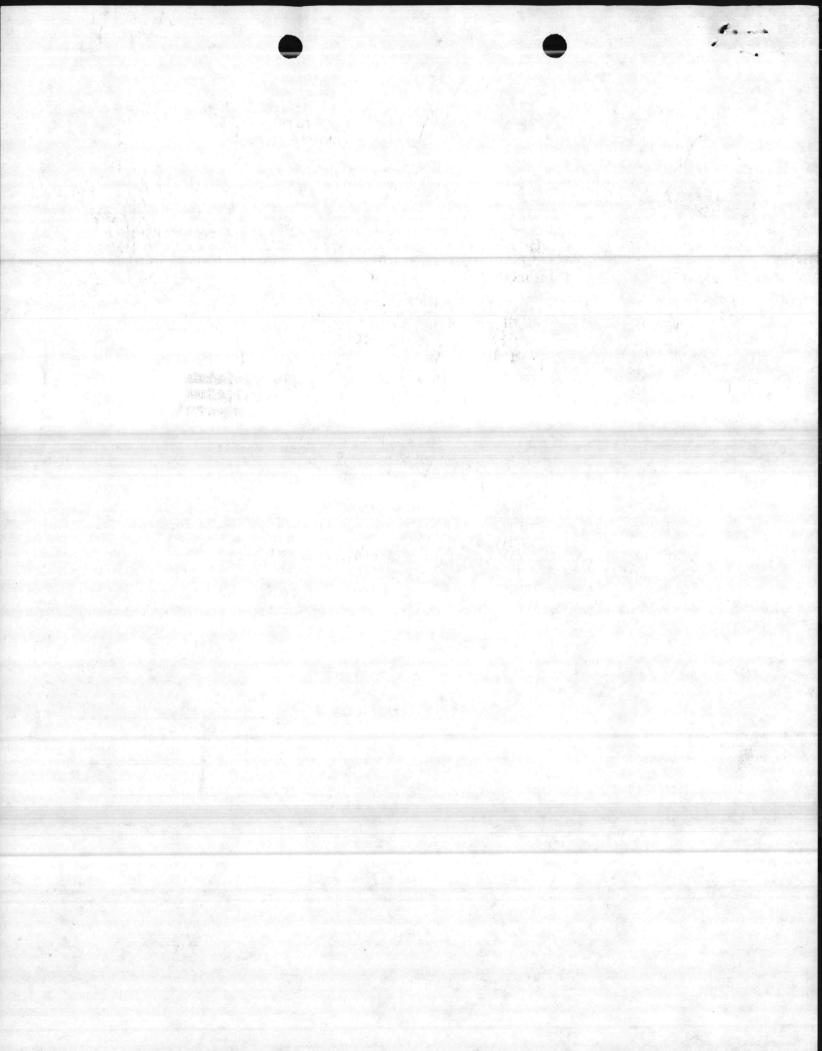
Return the ampul mailing container and a signed copy of this completed form to:

Dept. of Natural Resources & Community Development Division of Environmental Management Laboratory Branch P. O. Box 27687 Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code #_____Certificate #___

Laboratory reporting data Environmental Chemister and Michobiology LABORATORY Signature of laboratory supervisor Acadeth a Bay Date completed 15 December 1986

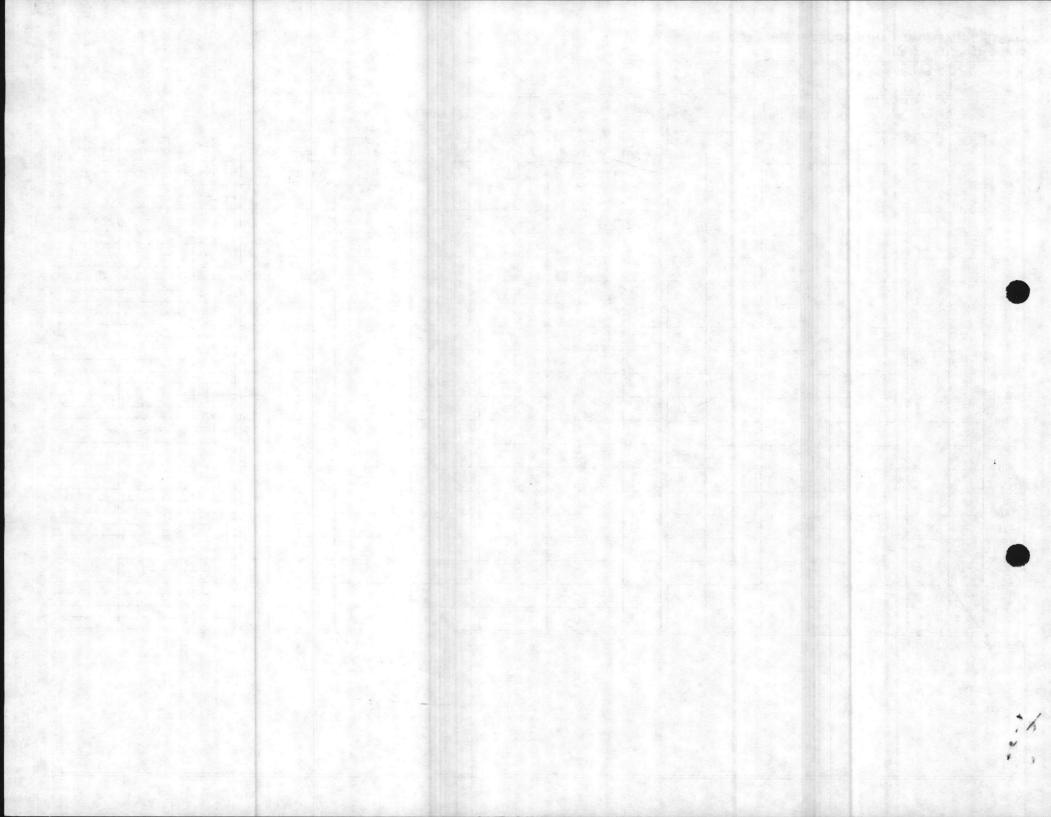


WASTE TREATMENT LABORATORY WORKSHEET

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BOD ELEMENT 00310					SUSPENDED SOLIDS ELEMENT 00530					COLIFORM							
PLANT		#	DO1	DO5	DEP	BOD mg/4	%	#	DISH & SOLID	DISH	SOLID	VOLUME	SOLIDS mg/l	%	DIL	TOTAL MFC/100 ML	FECAL MFC/100 MI
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REMARKS

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State of North Carolina Department of Natural Resources and Community Development

> Division of Environmental Management 512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary

December 9, 1986

R. Paul Wilms Director

Mr. Julian I. Wooten Director, Natural Resources Div Environmental Chemistry & Microbiology Laboratory Camp Lejeune, NC 28542

Dear Mr. Wooten:

Re: North Carolina Wastewater Laboratory Certification

We have received and reviewed your December 4, 1986, letter concerning laboratory certification. We have amended our files to reflect your current laboratory name and staff listing supplied with your letter. Attached is a report for the evaluation sample analysis performed by your laboratory. As shown on the report, we have enclosed Grease & Oil and total suspended residue rerun samples for your analysis. Also enclosed are initial evaluation samples for total phosphorus and ammonia nitrogen analysis. Acceptable results are required on all parameters prior to certification.

Thank you for the information supplied. Contact us at 919-733-3908 if you have questions.

Sincerely,

W. B. Edwards

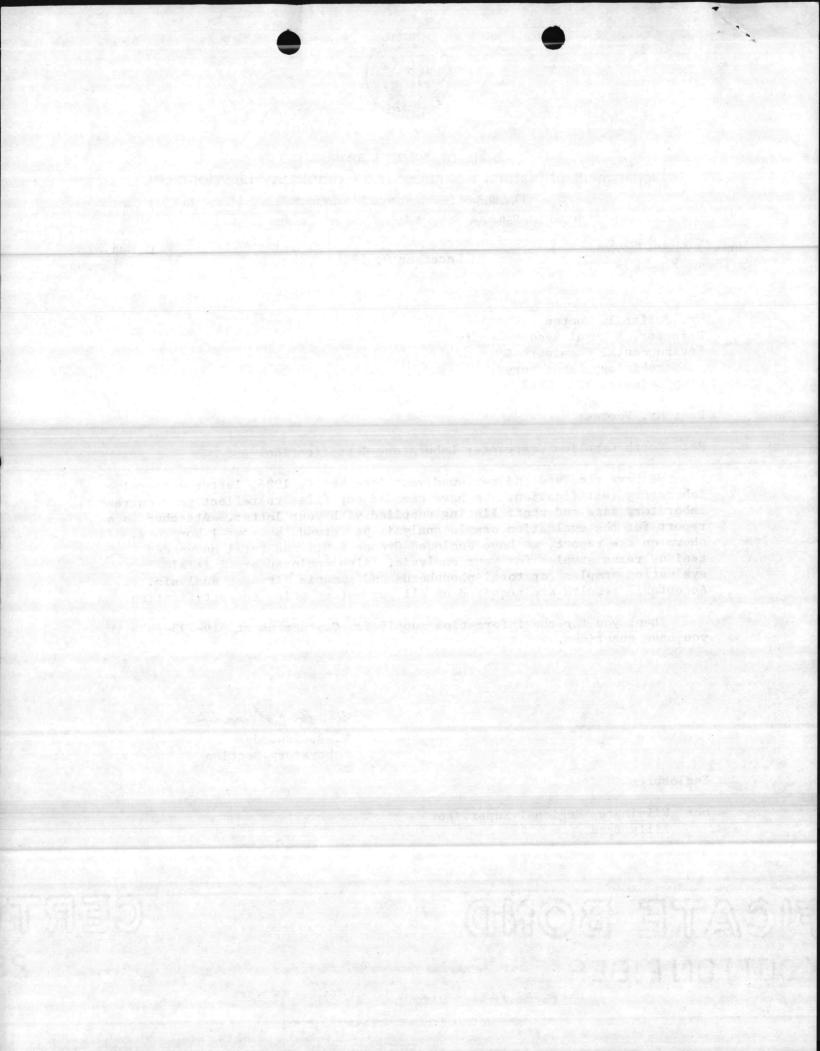
W. B. Edwards Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor Billy Byrd

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015







State of North Carolina Department of Natural Resources and Community Development

Division of Environmental Management

512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary

December 9, 1986

R. Paul Wilms Director

Mr. Julian I. Wooten Director, Natural Resources Div Environmental Chemistry & Microbiology Laboratory Camp Lejeune, NC 28542

Dear Mr. Wooten:

We have evaluated your results from the performance samples for wastewater laboratory certification received on <u>December 5, 1986</u>. The information from this evaluation is summarized below:

Parameter R	Value eported	True Value	State Lab Value	Acceptable Range	Performance	
BOD mg/1	33	38.65		28 - 49	Acceptable	
*Grease & Oil mg/1	4.0	26		19 - 33	Unacceptable	
pH Units	4.02	4.01		3.71-4.31	Acceptable	
*Suspended Residue	3 mg/1	30.5		22 - 39	Unacceptable	

() Your laboratory will be recommended for (certification) (recertification).

() You will be contacted to establish a date for your on-site laboratory inspection.

(*) Another sample(s) has been included for your use as required.

() You will be provided another sample at a future date.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

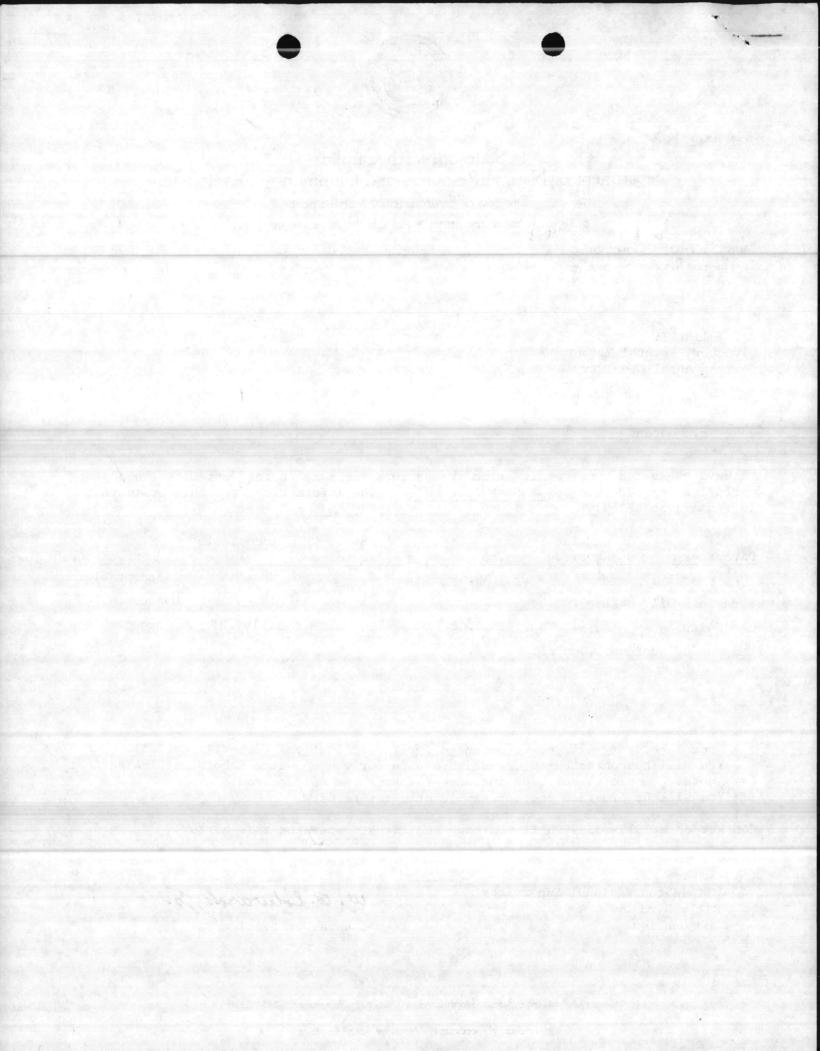
cc: Wilmington Regional Supervisor Billy Byrd Ted Cashion

W. B. Edwards, Js.

Laboratory Section

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



Besty

6740/3 NREAD(L) DEC 0 4 1986

Mr. W. B. Edwards NCRD/DEM, Laboratory Branch Post Office Box 27687 Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 15 October 1986 letter, the Certified Laboratory Performance Analysis Report is submitted. Your letter was received on 5 November 1986.

Since the 29 May 1985 submission of our application for Wastewater Analytical Laboratory Certification, several changes have occurred. The name of the laboratory has changed from the Water Quality Control Laboratory to the Environmental Chemistry and Microbiology Laboratory (ECML). There have been some changes in the laboratory staff and enclosed is a list of the present laboratory staff. showing their education and experience. The Base has also received their proposed National Pollutant Discharge Elimination System permits which show additional monitoring requirements for Ammonia, Total Nitrogen, Total Phosphorus, Temperature, and Dissolved Oxygen. The temperature and dissolved oxygen readings will be run by the Wastewater Treatment Plant operators under the direction of the ECML. The ammonia and total phosphorus analysis will be run by the ECML. The total nitrogen analysis is presently contracted through Applied Laboratory Services with Oxford Laboratories, Inc.

Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, AC/S, Facilities at (919) 451-5977.

Sincerely,

JULIAN I. WOOTEN Director, Natural Resources Division Assistant Chief of Staff, Facilities By direction of the Commanding General

DEC 0 1 1936

W. Call



Cert No

Gertified Laboratory Performance Analysis Report

This form is to be used to report the results of your analysis of the enclosed performance evaluation samples. One completed copy of this form is the <u>only report</u> needed. Do not return the individual report forms or mailing cartons. In the appropriate space below, enter the dilution made on the ampul contents (example: 5/1000 for 5 ml to 1000 ml), the value obtained from your analysis, and return this report to:

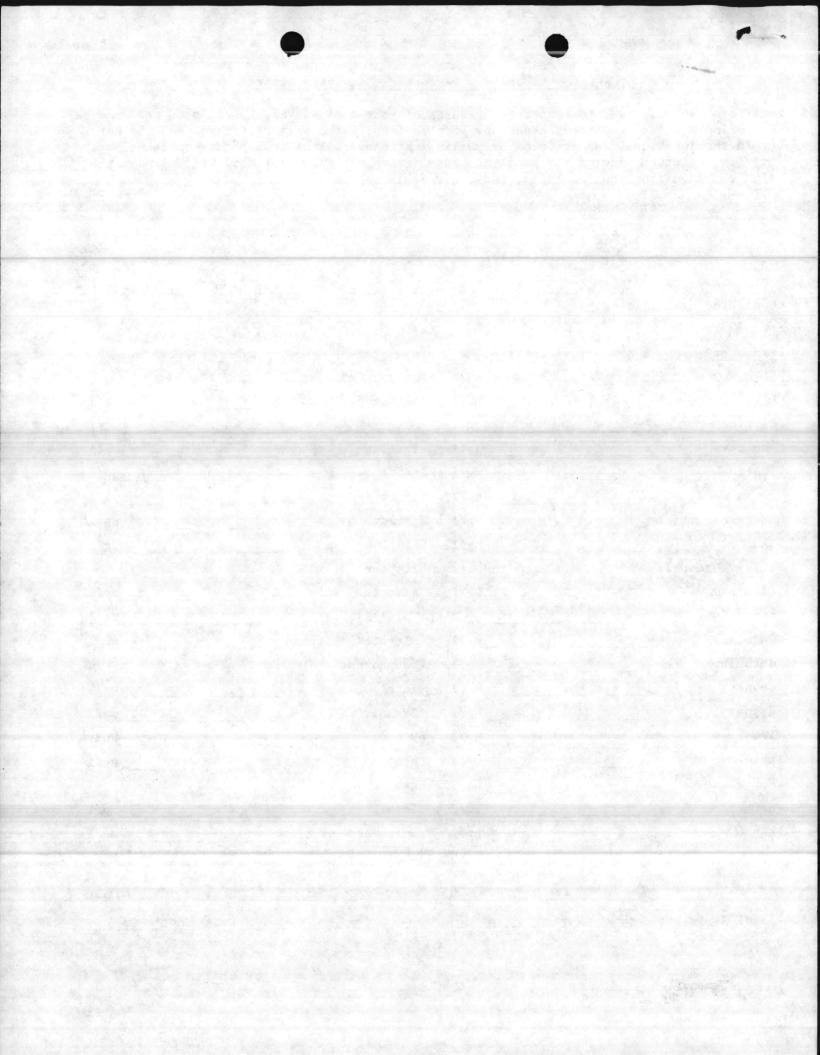
> NRCD/DEM Laboratory Section P. O. Box 27687 Raleigh, MC 27611-7587

within 30 days of your receiving date. Reports received after this 30 day period will be considered unsatisfactory.

Parameter	Dilution Made	Value Obtained	Parameter	Dilution Made	Value Obtaine	
BOD		mg/1	Metals, Group II:		the second second	
COD		mg/1	Antimony	the second		_µg/1
Chloride		mg/1	Silver			_µg/1
Cyanide		mg/1	Thallium	e de das		_µg/1
Fluoride		mg/1	Arsenic		No.	_µg/1
Grease & 011		4.0 mg/1	Barium			_µg/1
Hardness		mg/1	Mercury			_µg/1
MBAS		mg/1	Selenium			_µg/1
Metals, Group I:			Nitrogen:			
Aluminum		µg/1	Ammonia			_mg/1
Beryllium		µg/1	Total Kjeldahl			_mg/1
Cadmium		µg/1	Nitrate + Nitrite			mg/1
Chromium	1. 19.34	µg/1	Phosphorus:			
Cobalt		µg/1	Total (as P)			_mg/1
Copper	1	µg/1				mg/1
Iron		µg/1	PH		4.02	Units
Lead	in the second	µg/1				µg/1
Manganese		µg/1				
Nickel		µg/1				mg/1
Zinc	and the second	µg/1	Suspended		3	mg/1
			Turbidity			NTU

Laboratory reporting data ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LABORATORY

Signature Laboration Supervisor Date 12/2/86



ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LABORATORY STAFF

LABORATORY DIRECTOR - Elizabeth Betz

Education: High School, B.S. in Chemistry Certification: NCWWTPO Grade II, NCWTPO B-Well Experience: 7 years

TECHNICIANS:

Hoy J. Burns

Education: High School, 1 year College Certifications: Dept. of Health, Education & Welfare Clinical Laboratory Technologist, North Carolina Wastewater Operator Grade II Experience: Navy Clinical Laboratories - 11 yrs. Navy Research Laboratories - 9 yrs., Quality Control Laboratory - 10 yrs.

Carol Shores

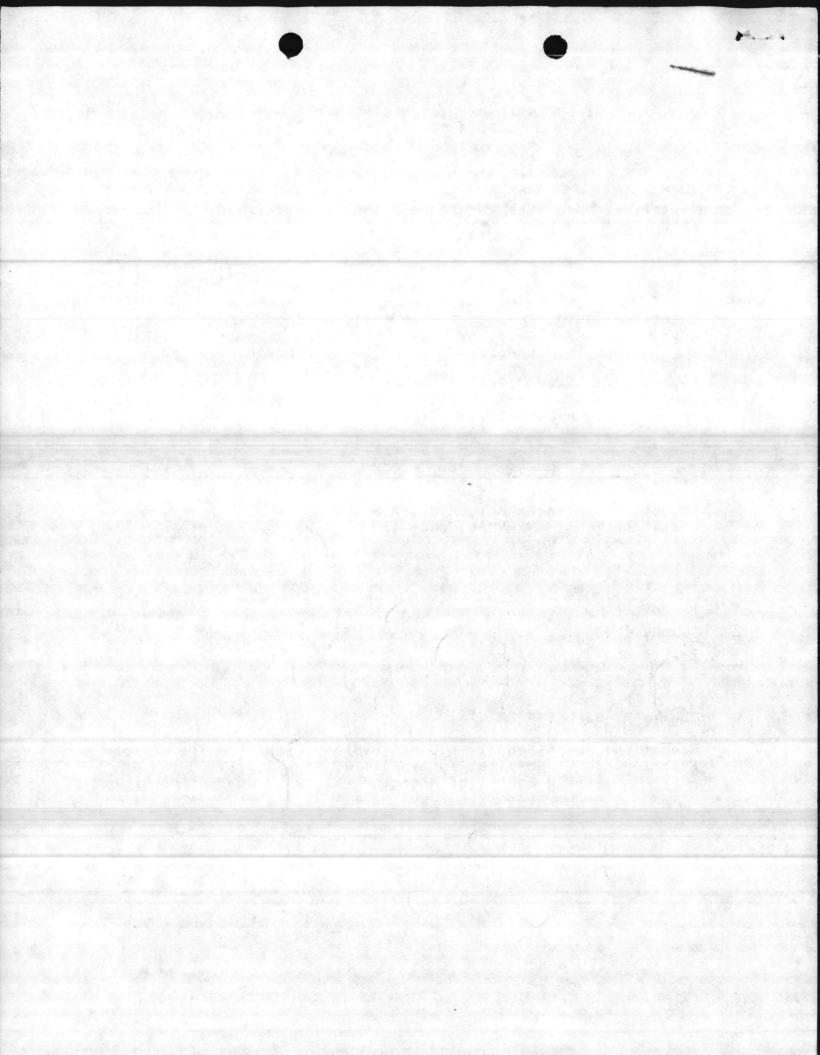
Education: High School, B.S., M.S. Certifications: NCWWTPO Grade II Experience: 2 years

Lyndia Lane

Education: High School Experience: 6 months

Thomas H. Barbee

Education: High School, B.S. in Biology with Wildlife Option Experience: VA Hospital in Asheville, NC - 2 yrs., ECU School of Medicine - 1 yrs., Quality Control Laboratory - 2 yrs.







State of North Carolina Department of Natural Resources and Community Development

Division of Environmental Management 512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary

October 15, 1986

R. Paul Wilms Director

Water Quality Control Laboratory Natural Resources & Environmental Affairs Div. MC Base Camp Lejeune, NC 28543

ATTENTION: Ms. Elizabeth A. Betz

Dear Laboratory Supervisor:

RE: Wastewater Laboratory Certification

In 1985 your laboratory submitted an application for Wastewater Laboratory Certification as required by NCAC 2H .0800. During the next few months, we will be contacting you to schedule a certification inspection of your laboratory. According to our records, your laboratory has not received and analyzed certification evaluation samples. Enclosed for your analysis are evaluation samples for parameters included in your certification application. Excluding pH, the samples are in concentrate form and must be diluted to prepare the actual sample for your analysis. Instructions are included that describe how each sample is to be diluted and reported. Within 30 days of receipt, please analyze these samples and report results to the address shown on the report form.

Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

W. B. Edwards

W. B. Edwards Laboratory Section

cc: Wilmington Regional Supervisor Mr. Billy Byrd

Enclosure

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015

An Equal Opportunity Affirmative Action Employer

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In 19.5 your labor for, subsite ' an application for Sartewated salutation (a.1) for the equivalence of a single formula for the equivalence of the second secon

Genteer va et 219-732 2006 17 yau ave questions of peak addith. Conveitor Instruction and Reporting Form for the Biochemical and Chemical Oxygen Demand Analyses

Please verify that the ampul you received corresponds with the number listed below:

D 102886

The contents of the enclosed ampul will be used to prepare two separate samples. Both samples should be formulated using clean dry volumetric pipets to transfer the specified amount of concentrate to separate volumetric flasks. Make each to volume with good quality distilled or deionized water and proceed immediately with the analyses.

Biochemical Oxygen Demand Sample: Transfer 10.0 ml of the ampul contents to a l liter volumetric flask and bring to volume with distilled or deionized water. Mix the solution thoroughly to obtain representative portions for analysis. This sample <u>must</u> be seeded and diluted by approved procedures to obtain a proper evaluation.

Chemical Oxygen Demand Sample: Transfer 5.0 ml of the ampul contents to a 250 ml volumetric flask and bring to volume with distilled or deionized water. Mix the solution thoroughly to obtain representative portions for analysis. This sample is now ready for analysis.

See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Perform Analyses Marked (X)	Analysis	Concentration Range	Report Results	Value Obtained
X	BOD	10 - 200 mg/1	to 1.0 mg/1	33 mg/e.
	COD	1.0 - 200 mg/l	to 1.0 mg/1	

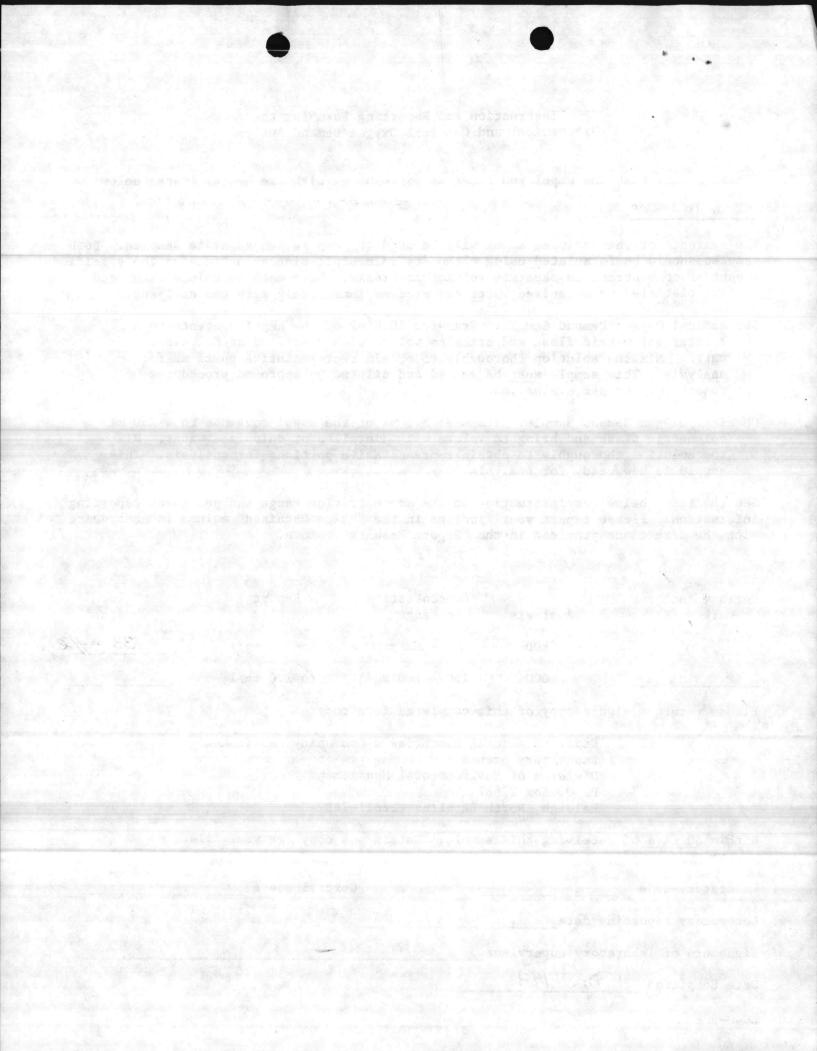
Please return a signed copy of this completed form to:

Dept. of Natural Resources & Community Development Laboratory Branch Division of Environmental Management P. O. Box 27687 Raleigh, North Carolina 27611-7687

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code	Certificate #
Laboratory reporting data	
Signature of laboratory supervisor	
Date Completed 1 DEC 1986	

LC40-8/84



Instruction and Reporting Form for Analysis of Oil and Grease

Please verify that the number on the ampul you received corresponds with the number listed here: G_{10} 28 86 . 10 mL/500 Vol.

The contents of this ampul are to be used to prepare a sample for the analytical performance test for oil and grease analysis. Add 500 ml of distilled or deionized water and 2.5 ml concentrated HCl to a separatory funnel. Transfer 5.0 ml of the ampul contents to the separatory funnel. Complete the analysis as detailed in the required procedure. Calculate the oil or grease concentration using the formula below:

mg/l grease or oil = (mg oil or grease in sample - mg oil or grease in blank) x 1000 500

Please record your result in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Perform analyses Marked (X)	Analysis	Concentration Refer	Report Results	Value Obtained
x	Oil & Grease	1-1000 mg/1	to 1 mg/1	4.0

Return the ampul mailing container and a signed copy of this completed form to:

Dept. of Natural Resources & Community Development Division of Environmental Management Laboratory Branch P. O. Box 27687 Raleigh, North Carolina 27611

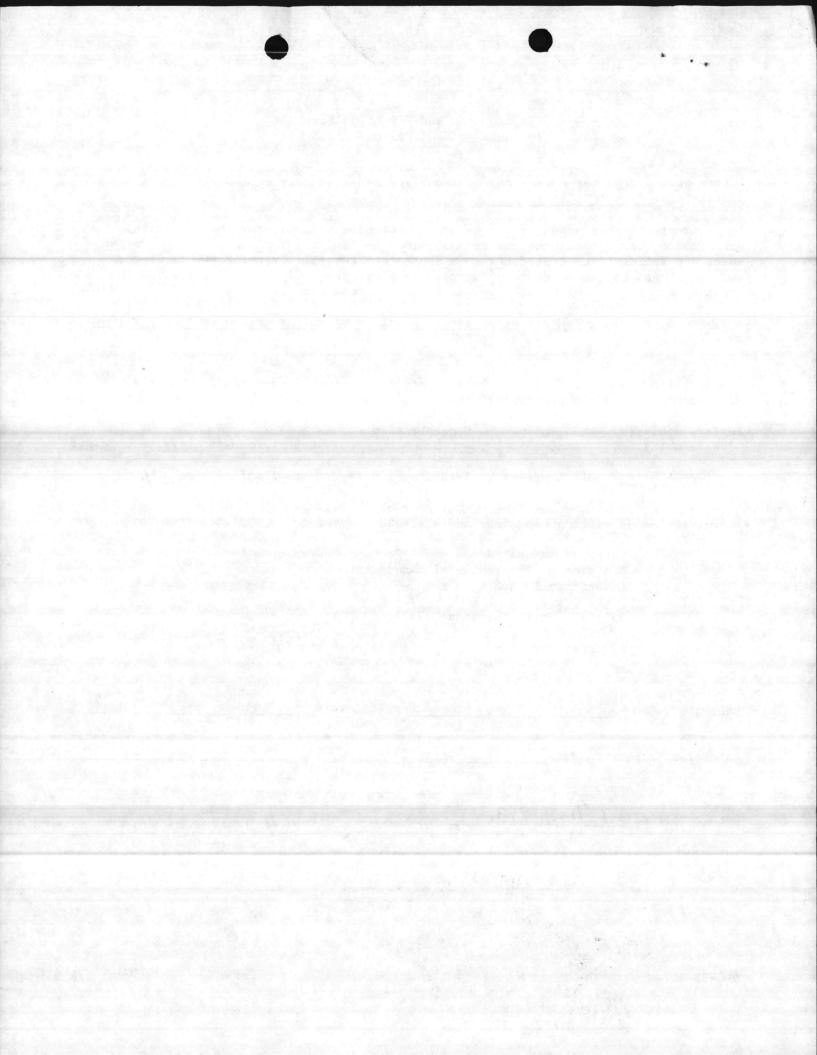
within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code #_____Certificate #

Laboratory reporting data

Signature of laboratory supervisor

Date completed 1 Dec 1996



Instruction and Reporting Form for Analysis of Total and Suspended Residue

Please verify that the ampul you received corresponds with the number listed below: R 10 28 86

The contents of this ampul are to be used to prepare an analytical performance sample for total and suspended residue analysis.

When you are ready to perform the analysis, remove the rubber seal from the vial containing the solids, being careful not to lose particles clinging to the rubber seal.

Pour the contents of the vial through a glass funnel into the 1000 ml volumetric flask. Use deionized water to quantitatively rinse the solids from the vial and rubber seal into the volumetric flask. Rinse the glass funnel by adding deionized water to make the sample up to the 1000 ml mark.

Mix well and analyze by standard procedures.

NOTE: Some labs have found it necessary to correct for their distilled water residue content when analyzing this type of sample.

See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

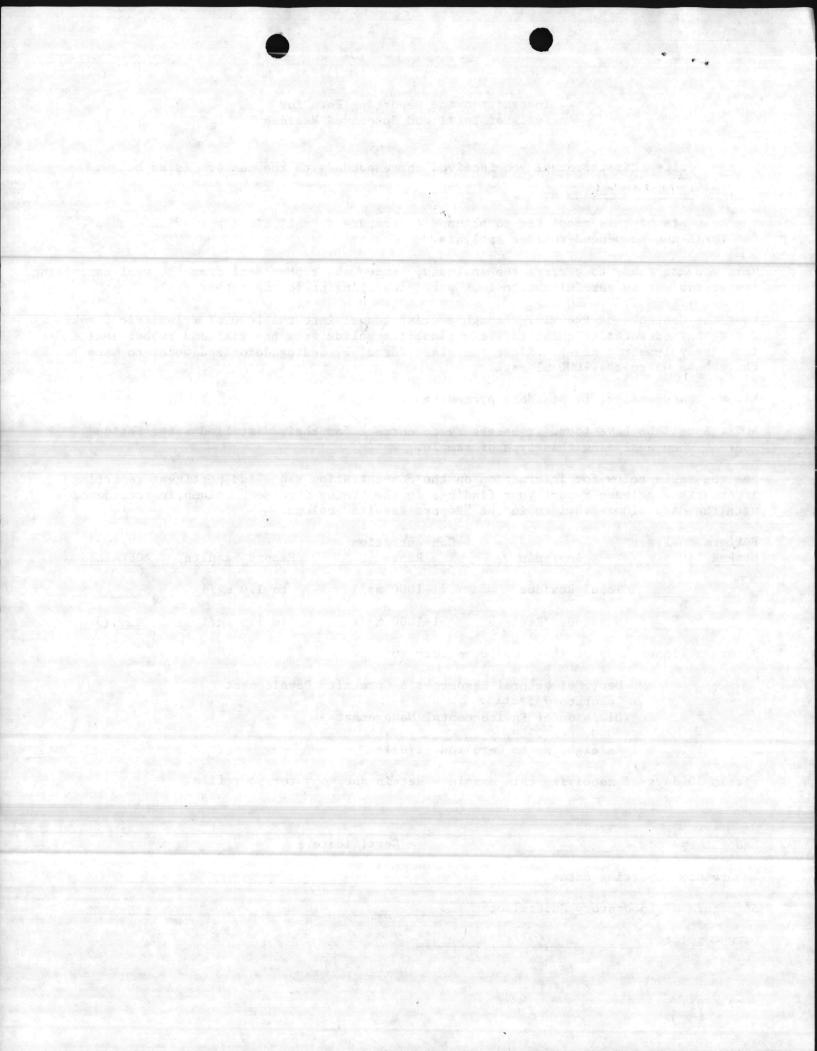
Perform Marked	analyses (X)	Analysis	Concentration Range	Report Results	Value Obtained
		Total Residue	1-1000 mg/1	to 1.0 mg/1	and the second
	Χ	Suspended Residue	1-1000 mg/1	to 1.0 mg/1	3.0

Return a signed copy of this completed form to:

Dept. of Natural Resources & Community Development Laboratory Section Division of Environmental Management P. O. Box 27687 Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code #	Certificate #
Laboratory reporting data	
Signature of Laboratory Supervisor	
Date Completed Z DEC 1986	



Instruction and Reporting Form for Analysis of pH

Please verify that the number on the ampul you received corresponds with the number listed here: pH 10 2886

To begin the analysis, open the ampul by snapping the top off at the narrow part of the neck and pour the contents directly into a 50 ml beaker. DO NOT DILUTE THIS CONCENTRATE. The sample is now ready for analysis.

Please record your result in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Analysis	Range	Report Results	Value Obtained
рН	0 - 14 Units	To 0.01 Units	4.02

Return a signed copy of this completed form to:

W. B. Edwards, Jr. NRCD/DEM Laboratory Section P. O. Box 27687 Raleigh, NC 27611-7687

within 30 days of receiving this sample. One copy for your file may be retained.

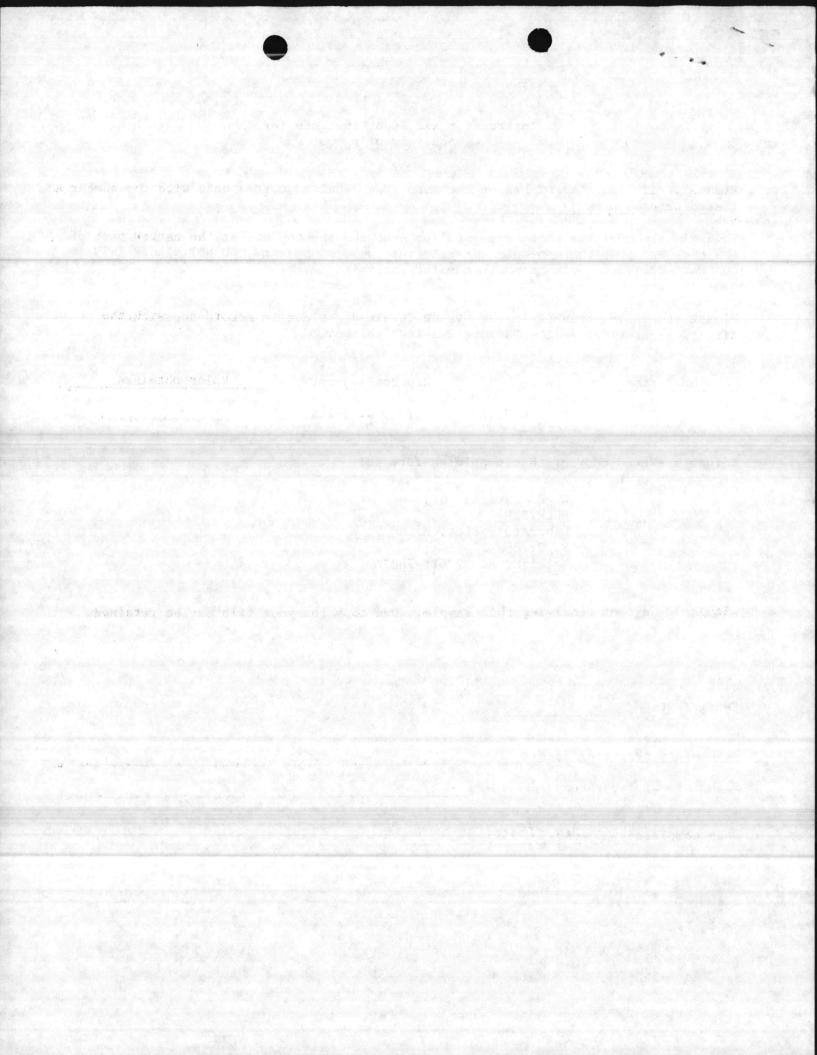
Certificate #

Laboratory reporting data

Signature of laboratory supervisor

Date completed Z Dec 1984

LC-15 (Rev.5-86)





6240 NREAD 29 May 1985

Mr. W. B. Edwards, Jr. NCRD/DEM, Laboratory Branch Post Office Box 27687 Raleigh, North Carolina 27611

Dear Sir:

The enclosed application for Wastewater Analytical Laboratory Certification is submitted in accordance with your letter of April 5, 1985. Our present National Pollutant Discharge Elimination System permit number is NCO003239. It is our understanding that your agency has assigned a separate number to each of our seven Sewage Treatment Plants. This application satisfies requirements applicable to the Camp Geiger Sewage Treatment Plant (NCO003239); Tarawa Terrace Sewage Treatment Plant (NC0020923), and Hadnot Point Sewage Treatment Plant (NC0020877).

Except as shown in Section VII 2 of the enclosed form, all laboratory analyses are performed by the Water Quality Control Laboratory, Natural Resources and Environmental Affairs Division, Marine Corps Base, Camp Lejeune. If additional information is desired, please contact Ms. Elizabeth A. Betz, Supervisory Chemist at (919) 451-5977.

Sincerely,

J. I. WOOTEN Director

Blind copy to:

Writer: D. Sharpe, NREAD 5003 Typist: J. Cross 29May85



STATE OF NORTH CAROLINA DEPARTMENT OF NATURAL RESOURCES & COMMUNITY DEVELOPMENT DIVISION OF ENVIRONMENTAL MANAGEMENT

APPLICATION FOR

WASTEWATER ANALYTICAL LABORATORY CERTIFICATION

I. Name of Laboratory (1) Water Quality Control Laboratory

Mailing Address	Natural Resources	s & Environmental Affairs I	Div., MCBase
		Street or Box No.	
	Camp Lejeune,	NC	28542
	City	State	Zip
Location	65 Molly Pitcher	Drive, Hadnot Point Area	
and the second second	Camp Lejeune	Street or Box No. North Carolina	28542
	City	State	Zip
Telephone Number	r (919)-451-5977		

II. Laboratory Personnel

A. Laboratory Supervisor Elizabeth A. Betz, Supervisory Chemist

1. Education - Give your complete education history below:

Penncrest High School	Media, PA 19063	Mo. June yr. 1975
High School Name	Location	Date Graduated

Education Beyond High School	Name & Location	Credits (Sem. or Qtr.Hrs)	Last Yr. Attended	Degree or Diploma & Yr. Rec'd	Major Subject
College or University	High Point College	Quantity sufficient for Degree	1979	BS	Chemist
Graduate or Professional					
Other Education, Internship, etc.					

List fields of work for which you are licensed, registered, or certified, giving date(s) and sources(s) of issuance. Wastewater Operator, Crade II

N. C. Water Treatment Operator B-Well

(1) If this is a wastewater treatment plant (WWTP) with no onsite laboratory, enter the WWTP name and address and complete sections VII and VIII only.

Date 29 May 1985

Laboratory Supervisor (nont.)

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Page 2.

3

	Date employed Nov 1979	Full-Time X	Years 5	Months 6	
	No. employees supervised by	Part-Time	Years	_ Months	-
	уоц _4	(If part-time n	no. hours pe	er week)
	Employer Camp Lejeune MCBase	Address Camp	Lejeune,	NC 28542	-
	Duties Supervise laboratory;	responsible f	for monito	ring Water	
	Treatment, Wastewater Treat	ment and Hazar	dous Wast	e	-
					•
(Ъ)	Title of next to last position	N/A			-
	Date employed	Full-Time	Years	Months	-
	Date Separated	Part-Time	_Years	Months	-
	No. employees supervised by you	(If part-time	no. hours p	per week)
	Name and title of supervisor				-
	Employer	Address			-
	Duties				
					-
		· · · ·			-1
	Title of next position N/A				-
	Date employed	And a second state of the	The second second	Months	-
		Part-Time	Years	Months	-
	Date Separated				
	No. employees supervised by you	(If part-time	no. hours	per week	2
	No. employees supervised by you	and the second second	ing ngagalini	per week	-
	No. employees supervised by you Name and title of superviser Employer	(If part-time Address	ing ngagalini	per week	-
	No. employees supervised by you	and the second second	ing ngagalini	per week	
	No. employees supervised by you Name and title of superviser Employer	and the second second	ing ngagalini	per week	
	No. employees supervised by you Name and title of superviser Employer Duties	Address			
3.	No. employees supervised by you Name and title of superviser Employer Duties References - persons familiar wit	_ Address h your professio	nal compete	ncy:	
3.	No. employees supervised by you Name and title of superviser Employer Duties References - persons familiar wit (a) Danny Sharpe, Supervisory	Address h your profession Ecologist, P(nal compete O Box 743,	ncy: , Richlands, I	
3.	No. employees supervised by you Name and title of superviser Employer Duties References - persons familiar wit (a) Danny Sharpe, Supervisory (b) Julian Wooten, Dir, NREAD	Address	n al compete O Box 743, s Base, Ca	ncy: , Richlands, Mamp Lejeune, M	10
	No. employees supervised by you Name and title of superviser Employer Duties References - persons familiar with (a) Danny Sharpe, Supervisory (b) Julian Wooten, Dir, NREAD (c) Don Beesley, LabCert. Eva	Address	n al compete O Box 743, s Base, Ca	ncy: , Richlands, Mamp Lejeune, M	10
3.	No. employees supervised by you	Address h your profession Ecologist, Po Marine Corps aluator, DHS, 1	nal compete O Box 743, s Base, Ca PO Box 280 Years o	ncy: , Richlands, 1 amp Lejeune, 1 047, Raleigh, f Analytical	10
	No. employees supervised by you	Address	nal compete O Box 743, s Base, Ca PO Box 280 Years o	ncy: , Richlands, Mamp Lejeune, Mamp Lejeune, Manager, Ma	VIC
	No. employees supervised by you	Address	nal compete O Box 743, s Base, Ca PO Box 280 Years o	ncy: , Richlands, 1 amp Lejeune, 1 047, Raleigh, f Analytical	<u>1</u> C
	No. employees supervised by you	Address	nal compete O Box 743, s Base, Ca PO Box 280 Years o	ncy: , Richlands, 1 amp Lejeune, 1 047, Raleigh, f Analytical	VIC

B. Other Laboratory Personnel

Hoy J. Burns

Education: High School, 1 year College

Certifications: Dept. of Health, Education & Welfare Clinical Laboratory Technologist, North Carolina Wastewater Operator Grade II

Experience: Navy Clinical Laboratories - 11 yrs. Navy Research Laboratories - 9 yrs. Quality Control Laboratory - 9 yrs.

Robert J. Lachapelle, 2 yrs. College, Navy Medical Technologist School Certifications: Certified Medical Technologist

Experience: Navy Laboratories - 17 yrs. Quality Control Laboratory - 4 yrs

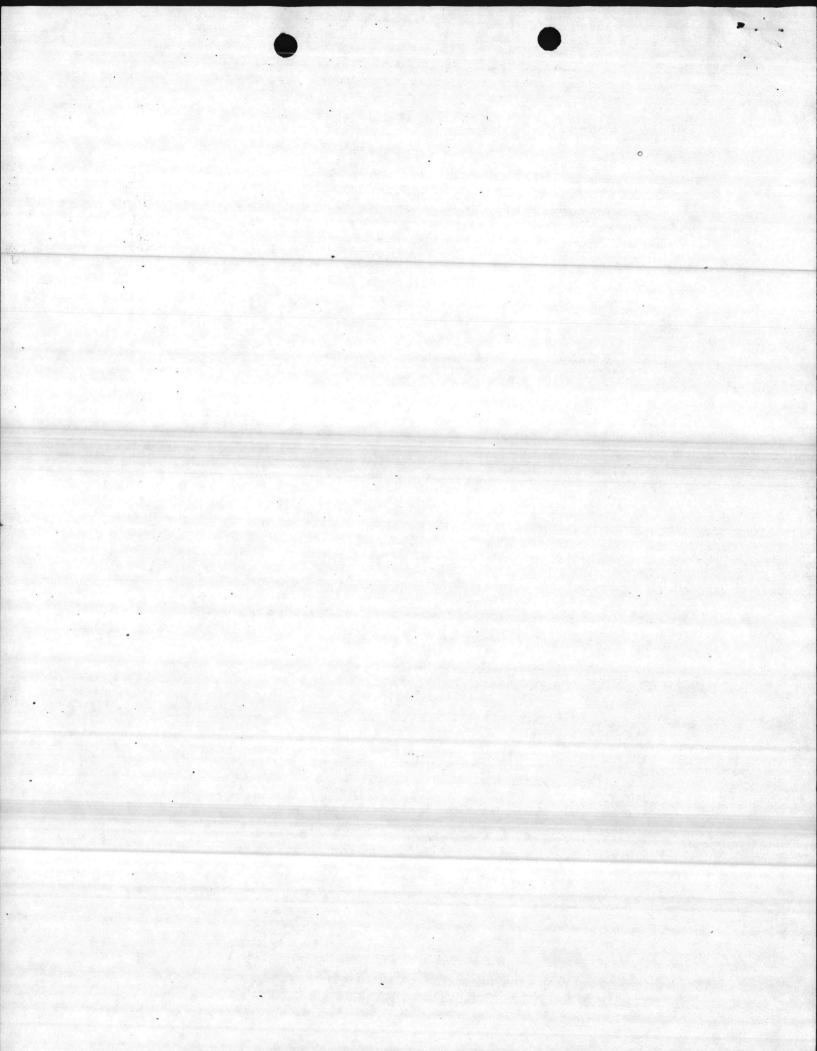
Gaines B. Huneycutt, Jr.

Education: High School, AAS in Fish & Wildlife Management Certifications: North Carolina Water Treatment Operator Grade C-Well

Experience: N.C. Department of NatRes & ComDev- 6.5 yrs. Contracts & Engineers Services - 1 yr; Quality Control Laboratory - 4 yrs.

Thomas H. Barbee

Education: High School, BS in Biology with Wildlife Option
Experience: VA Hospital in Asheville, NC - 2 yrs;
ECU School of Medicine - 1 yr. Quality Control Laboratory - 1 yr.



Page 3.

Parameters presently identi.

III. Parameters for which certification may be requested: fied in our Permit Laboratories need to obtain certification only for those parameters which will be reported to the state in compliance with monitoring and pretreatment regulations. Please (1) circle your choice of analytical method in each instance from the list to the right of the parameter, and (2) cite your usual minimum reporting concentration for it to the far right.

		Refer	ence	a hand a second light	
Analytical Parameter	1979 EPA Methods	15th Ed. Standard Methods	Others Include Reference & Methods	Min. Report Concentration (incl. Units)	
BOD	405.1	(507)		l mg/L	
COD	410.1	508A			
	410,2				
	410.3				
	410.4				
Chloride	325.3	407A			
	325.2	407B		and the second	
	325.1	407D			
Coliform, Fecal MF	124 ⁽¹⁾	909C		1/100 ML	
Coliform, Total MF	108 ⁽¹⁾	(909A)		1/100 ML	
Coliform, Fecal Tube	132 ⁽¹⁾	(908C)		<4/100 ML	
Coliform, Total Tube	114 ⁽¹⁾	(908A)		(4/100 ML	
Cyanide	335.2	412B			
	335.3	412C		and the second	
		412D		and the second second second second	
Fluoride	340.2	413Å			
	340.1	413B	•		
	340.3	413C			
		413E		and the second second	
Grease and Oil	413.1	(503A)	1 Martin Carl	l mg/L	
Hardness, Total	130.1	314B			
	130.2	314A			
	215.1+ 242.1			and the states of the second	
MBAS	425.1	512A			

(1) Coliform procedures taken from "Microbiological Methods for Monitoring the Environment", 1978, EPA-600/8-78-017.

	Reference				
Analytical Parameter	1979 EPA Methods	15th Ed. Standard Methods	Others Include Reference & Methods	Min. Report Concentration (incl. Units)	
Metals Group I					
Aluminum	202.1	303C	an a		
	202.2	304			
e se		306B			
Beryllium	210.1	303C			
	210.2	304			
		309B			
Cadmium	213.1	303A			
	213.2	303B			
		304			
		310B			
Chromium, Total	218.3	303A			
	218.1	303B			
	218.2	304			
		312A			
Cobalt	219.1	303A	S Children Mound , and other	In the second second	
	219.2	303B			
		304			
Copper	220.1	303A			
	220.2	303B			
		313B			
Iron	236.1	303A			
	236.2	303B			
William and a start		304			
		315B			
Lead	239.1	303A			
	239.2	303B			
		304			
and the second		316B	All and a second se		
Vengenege	243.1	303A			
Manganese	243.2	303B			
		304			

319B

Page 4.* • :

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Analytical Parameter	1979 EPA Methods	15th Ed. Standard Methods	Others Include Reference & Methods	Min. Report Concentration (incl. Units)
Nickel	249.1	303A		
	249.2	303B		
		304		second strange sector
		321B		
Zinc	289.1	303A		A State of the second
	289.2	303B		1 4 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
		304		
Metals Group II				
Antimony	204.1	303A		
and the second	204.2	304		
Silver	272.1	303A	10 10 10 10 10 10 10 10 10 10 10 10 10 1	
	272.2	303B		
		304		
Thallium	279.1	303A		
anger og som en sen er som en sen er som er som Den som er so	279.2	304		
Arsenic	206.5	303E		
	206.3	304		
· · · ·	206.2	307B		and the second second
	206.4			
Barium	208.1	303C		
· · · · · · · · · · · · · · · · · · ·	208.2	304		
Mercury	245.1	303F		-
	245.2			
Selenium	270.2	304	A STATE OF	
	270.3	303E		
Ammonia, Nitrogen	350.2	417A	(1) In the Source State and Source State and Source State and Source State Stat State State State State State S	
	350.3	417B		i i i i i i i i i i i i i i i i i i i
	350.1	417D		
		417F		
Total Kjeldahl Nitrogen	351.3	420A		
(TKN)	351.1	420B	an en	
	351.2	_417D		
	351.4	417B		
 m = Stand in Mir and in the in the second sec		417E		

		Reference			
Analytical Parameter	1979 EPA Methods	15th Ed. Standard Methods	Others Include Reference & Methods o	Min. Report Concentration ds o (incl. Units	
Nitrate plus Nitrite	353.3	418C		far - ballet file	
Nitrogen	353.2	418F			
	353.1				
Total Phosphorus	365.2	424C (111)			
•	365.3	424F		•	
	365.1	424G			
	365.4				
Orthophosphate	365.1	424G			
	365.2	424F			
	365.3				
рН	150.1	423		0.1 Unit	
Phenols	420.1	510A ⁽²⁾			
		510B ⁽²⁾			
	420.2	510C ⁽²⁾			
Residue, Total	160.3	209A			
Residue, Suspended	160.2	209D		l mg/L	
Turbidity (2)	180.1	214A			

(2) Standard Methods, 14th Edition

IV. Laboratory Test Equipment

Instrument	Manufacturer/Model	Used in Analysis For:
pH/Dissolved Oxygen Meter	Orion 611	Dissolved Oxygen pH
Oven .	Fisher Model 215F	Solids
Coliform Incubator Bath	GCA/Precision Scientific	Fecal Coliform MF
Dry Air Incubator	Elconap	Total Coliform MF
Incubator .	Precision Model 815	BOD
Autoclave	Market Forge Sterilmatic	Sterilizing Micro Equipment
Dry Air Incubator	Precision Model 2	Total & Fecal Coli M
Oven	Fisher Model 438F	Oil & Grease
Water Bath	Precision	Oil & Grease
Shaker	Kraft Model S-500	Oil & Grease
Analytical Balance	Mettler H33	Solids & Oil & Greas
Vacuum Pump	Precision	Solids, Coliform
the second s		and the second

- V. Quality Control Program
 - Does your laboratory have a written quality assurance document? Yes (X) No ().
 - Does your laboratory analyze duplicate samples daily for each parameter? Yes (X) No ().
 - 3. Does your laboratory analyze known standards for each parameter each day samples are analyzed? Yes () No (X).
 - 4. Does your laboratory meet the requirements of Table II Required Containers, Preservation Techniques, and Holding Times as shown in the October 26, 1984, Federal Register? Yes (X) No (). If no, enter any deviations.

 Briefly summarize your analytical quality assurance procedures for the parameters which certification has been requested. Use additional pages if necessary.

See attached pages

- VI. Laboratory Type
 - 1. Is your laboratory applying for certification as a commercial laboratory? Yes () No (X)
 - Is your laboratory applying for certification as a municipal or industrial laboratory? Yes (X) No ().
 - 3. Is your laboratory performing wastewater analysis for clients in North Carolina? Yes (X) No ()
 - Does your laboratory wish to seek clients in North Carolina? Yes () No (X).

VII. Municipal and Industrial Wastewater Treatment Plants Only

- 1. Enter your NPDES Permit Number NC0003239; NC002087.7 and NC0020923
- 2. Are state required monitoring analyses for this facility performed by another laboratory? Yes () No (X). If yes, give the name and address of the laboratory and the parameters performed by that laboratory.

NOTE: Plant personnel measure or test for flow and pH.

Remaining analyses are performed by Water Quality Control

Laboratory.

VIII. This statement certifies that the information contained in this application is truthful and accurate to the best of my knowledge.

Date 29 May 1985

Signature of Applicant ELIZABETH A. BETZ Supervisory Chemist Title Page 8

To apply for certification, return two copies of this application to:

W. B. Edwards, Jr. NRCD/DEM, Laboratory Branch P. O. Box 27687 Raleigh, N. C. 27611 FOR WATER QUALITY CONTROL LABORATORY, MARINE CORPS BASE, CAMP LEJEUNE NORTH CAROLINA:

V. 5. Analytical Quality Assurance Procedures

BOD (Biochemical Oxygen Demand)

Normally the BOD samples received by this laboratory are unchlorinated. The samples are screened for chlorine before processing. Seeding is, therefore, not usually required. The dissolved oxygen meter is calibrated against a Winkler Titration with each batch of samples. Dilution water blanks are set up with each batch of dilution water. When blanks have depletions greater than 0.2 mg/l, the corresponding batch of samples is discarded. A set of glucoseglumatic acid standards, with seeding, is set up weekly. Duplication is done daily on one set of samples.

Coliform, Total & Fecal, MF & MPN

Incubator temperatures are read daily. Negative (Pre and Post) and positive controls are run with each set of samples. Any samples but potable water are run with at least two dilutions. Duplicates are also run daily. The laboratory maintains N.C. Microbiology certification for drinking water and all applicable quality control on media, sterilization of equipment, etc.

Oil and Grease

A standard and blank are run with each batch of samples

pH

Meters are standardized using low and high buffers daily. Monthly, meters are checked against certified buffers.

Residue, Solids

Oven temperatures are recorded on Laboratory Worksheets. A duplicate is analyzed daily.

General

All balances are zeroed daily. Analytical balance is checked against a set of standard weights monthly. All chemicals are analytical reagent grade. All reagents are dated when prepared. Copies of the 1979 EPA Methods and 15th Edition of Standard Methods are maintained in the Laboratory. TON PATER OUNDE<mark>RT O</mark>NTEOL LABORATORY, UNTERT CORPERATORY, ATA AND ATA ON THE LABOR OF THE CORPERATORY

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Normally the samples are the set in effective there we use directively. The samples are the set in effective terms protors and leading in the relations for asually redukted. We detailed on an use if is rely reduktion water that be with each bits along of the set of the relation water that is the set of the set of the set of the relation of the set of the set of the concept of the set of each is iscarded. A set of elected relation is done dely in the set of relation.

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C. CULTOS . SULLBOR

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Professional Other Education, Internship, etc.

List fields of work for which you are licensed, registered, or certified, giving date(s) and sources(s) of issuance. WASTEWATER OPERATOR, GRADE TI

N.C. WATER TREATMENT OPERATOR B-WELL

Graduate or

(1) If this is a wastewater treatment plant (WWTP) with no onsite laboratory, enter the WWTP name and address and complete sections VII and VIII only.

Date

Laboratory Supervisor (cont.)

- 2. Experience:
- (a) Title of present position SUPERVISORY CHEMIST

Date employed 11/79	Full-Time	Years 5	Months 6
No. employees supervised by	Part-Time	Years	Months
you	(If part-time	no. hours pe	er week
Employer CARLE LEJEUNE MCB	Address Camp	LEJEUNE,	NC 2854
Duties SUPERVISE LABORATORY	REPONSIBLE I	DR MONIT	ORING
WATER TREATMENT WASTEWA	TER TREATMO	ENT AND	HAZARDOUS

1 A I	- ^	100 000	10 C	
VV	24	2	VE	1.8
(Second		Contraction of the local division of the loc	Supply and	-

b) Title of next to last position <u></u>	NIT	N. But Bar	a Articly of
Date employed	Full-Time	Years	Months
Date Separated	Part-Time	Years	Months
No. employees supervised by you	_ (If part-tim	ne no. hours	per week_
Name and title of supervisor			
Employer	Address		
Duties			

Date employed	Full-Time	Years	Months
Date Separated	Part-Time	Years	Months
No. employees supervised by you	(If part-tim	ne no. hours	per week
Name and title of superviser		her strengt and	d and and and
Employer	Address	and the second second	. And the second
mproyer			

3. References - persons familiar with your professional competency: (a) DANNY SHAVPE, Supervisory Ecologist, P.O.BOX 743, Richlands NC. 28574 (b) Julian WooTen, NREAD MArine Corps Base Cang Deseure 28542 (c) DON BEESLEY, LABCERT. Evoluter, DHS, P.O.Box 28047 Reley 27611

B. Other Laboratory Personnel

Name	Education	Laboratory Experience
SE	E ATTACHED SHE	ET

Page 2.

- Laboratory Type * VI.
 - Is your laboratory applying for certification as a commercial laboratory? Yes () No ()
 - Is your laboratory applying for certification as a municipal or 2. industrial laboratory? Yes (>>> No ().
 - Is your laboratory performing wastewater analysis for clients in 3. North Carolina? Yes (>) No ()
 - Does your laboratory wish to seek clients in North Carolina? 4. Yes () No ().

VII. Municipal and Industrial Wastewater Treatment Plants Only

- 1. Enter your NPDES Permit Number NC0003239, NC0020877, And NC 0020923 hat your orgener has assumed each
- 2. Are state required monitoring analyses for this facility performed by another laboratory? Yes () No 🚫. If yes, give the name and address of the laboratory and the parameters performed by that laboratory.

Note. Plant personnel measure or Test for flow and pH. Remaining Analyses are performed In water Quality Control Laboratory, 00 50

This statement certifies that the information contained in this application VIII. is truthful and accurate to the best of my knowledge.

Date

Signature of Applicant

and Bilon the

Application .

Shald be mailed N.L.T.

30 MAY 85

Title

To apply for certification, return two copies of this application to: Julian & accommend you sim cover letter

W. B. Edwards, Jr. NRCD/DEM: Laborato P. O. Box 27687 Raleigh, N. C. 27

1

Quality Control Program

v.

- Does your laboratory have a written quality assurance document? Yes (<) No ().
- 2. Does your laboratory analyze duplicate samples daily for each parameter? Yes No ().
- Does your laboratory analyze known standards for each parameter each day samples are analyzed? Yes. () No ().
- 5. Briefly summarize your analytical quality assurance procedures for the parameters which certification has been requested. Use additional pages if necessary.

SEE ATTACHED PIGES

- VI. Laboratory Type *
 - Is your laboratory applying for certification as a commercial laboratory? Yes () No (>)
 - Is your laboratory applying for certification as a municipal or industrial laboratory? Yes (>>> No ().
 - Is your laboratory performing wastewater analysis for clients in North Carolina? Yes (>>> No ()
 - Does your laboratory wish to seek clients in North Carolina?
 Yes () No (

VII. Municipal and Industrial Wastewater Treatment Plants Only

- 1. Enter your NPDES Permit Number NC0003239, NC002.0877, And NC 00 20923
- Are state required monitoring analyses for this facility performed by another laboratory? Yes () No O.
 If yes, give the name and address of the laboratory and the parameters performed by that laboratory.

Note. Plant personnel measure or Test for pH. Remaining Analyses low and DANTOMA an Quality Control Labora Water

VIII. This statement certifies that the information contained in this application is truthful and accurate to the best of my knowledge.

Date_____

Signature of Applicant

Title

To apply for certification, return two copies of this application to:

W. B. Edwards, Jr. NRCD/DEM, Laboratory Branch P. O. Box 27687 Raleigh, N. C. 27611

Quality Control Program

v.

- Does your laboratory have a written quality assurance document? Yes (<) No ().
- Does your laboratory analyze duplicate samples daily for each parameter? Yes No ().
- Does your laboratory analyze known standards for each parameter each day samples are analyzed? Yes. () No (X).
- 5. Briefly summarize your analytical quality assurance procedures for the parameters which certification has been requested. Use additional pages if necessary.

SEE ATTACHED PAGES

	and the second				
Analytical Parameter	1979 EPA Methods	Refe 15th Ed. Standard Methods	Others Include Reference & Methods	Min. Report Concentratio (incl. Units	
Nitrate plus Nitrite	353.3	418C		attender i	
Nitrogen	353.2	418F		- An The Alexandre Bar	
	353.1			a charactering	
Total Phosphorus	365.2	424C (111)		A AP I HAR AND	
	365.3	424F		the second second	
	365.1	424G			
	365.4		and a state of a second	investigation of the second	
Orthophosphate	365.1	424G	A LOUSE REV.	and the second	
	365.2	424F			
	365.3				
рН	150.1	423		OIL UNIT	
Phenols	420.1	510A ⁽²⁾		Har sector for the sec	
	A State Backbonn	510B ⁽²⁾			
	420.2	510C ⁽²⁾			
Residue, Total	160.3	209A			
Residue, Suspended	160.2	209D		Imall	
Turbidity (2) charles in the last 1/11	180.1	214A			

(2) Standard Methods, 14th Edition

IV. Laboratory Test Equipment

Instrument	Manufacturer/Model	Used in Analysis For:
PH / DISSONED OXYEN METER	ORION 611	DISSOLVED OXYGEN DH
OVEN	FISHER MODEL 215F	SOLIDS
COLIFORM INCUBATOR BATH	GCA/ PRECISION SCIENTIFIC	FECAL COLIFSEM MF
DRY AIR INCUBATOR	ELCONAP	TOTAL COLLEGEN MA
INCURATOR	PRECISION MODEL 815	BOD
AUTOCLAVE	MARKET BREE STERLIMATIC	STERILIZING MICRO EUDIPMENT
Der AIR INCUBATOR	PRECISION MODEL 2	TOTAL + FECAL COLI MPN
OVEN	FISHER NODEL 438F	OIL + GREASE
WATER BATH	PRECISION	OIL + GRENSE
SHAKER	KRAFT MODEL S-500	OIL+ GREASE
ANALYTICAL BALANCE	METTLER H33	SOLIDS + OIL + GERASE
VACUUM RUMP	PRECISION	SUNDS, COLIFORM
	a province the second	

Analytical Parameter				
	1979 EPA Methods	15th Ed. Standard Methods	Others Include Reference & Methods	Min. Report Concentration (incl. Units)
Nickel	249.1	303A		
	249.2	303B		
		304		
		321B		
Zinc	289.1	303A		
	289.2	303B		
		304		
Metals Group II				and an agen in
Antimony	204.1	303A		
	204.2	304		
Silver	272.1	303A		
	272.2	303B		and the second second
		304		
Thallium	279.1	303A		
	279.2	304		C
Arsenic	206.5	303E		
	206.3	304	and the second second	and a start age of
	206.2	307B	and the second	
	206.4			
Barium	208.1	303C		
	208.2	304		
Mercury	245.1	303F		
	245.2	2. J E.		
Selenium	270.2	304		and the second second
	270.3	303E	2	
Ammonia, Nitrogen	350.2	417A		
	350.3	417B		
	350.1	417D		Property and
		417F		
Total Kjeldahl Nitrogen	351.3	420A		
(TKN)	351.1	420B	And the second	a series and the series
	351.2	417D		A Maria Street
	351.4	417B		
		417E		141

III. Parameters for which certification may be requested: IN OUR PERMIT Laboratories need to obtain certification only for those parameters which will be reported to the state in compliance with monitoring and pretreatment regulations. Please (1) circle your choice of analytical method in each instance from the list to the right of the parameter, and (2) cite your usual minimum reporting concentration for it to the far right.

PARAMETERS PRESENTLY IDE

Page 3.

FIEN

Analytical Parameter				
	1979 EPA Methods	Refer 15th Ed. Standard Methods	Others Include Reference & Methods	Min. Report Concentratio (incl. Units
BOD	405.1	507		1 mg/2
COD	410.1	508A		5
	410.2			
	410.3			
	410.4		and a second strange of the second strange of the second second second second second second second second second	
Chloride	325.3	407A		
	325.2	407B		and a start of the Desider
	325.1	407D		
Coliform, Fecal MF	124 ⁽¹⁾	(909C)		1/100 mL
Coliform, Total MF	108 ⁽¹⁾	(909A)		1/100 ML
Coliform, Fecal Tube	132 ⁽¹⁾	908C		24/100 mc
Coliform, Total Tube	114 ⁽¹⁾	908A		= 4/100 mc
Cyanide	335.2	412B		
	335.3	412C		
		412D		a desta de la companya de la company La companya de la comp
Fluoride	340.2	413A		
	340.1	413B		
	340.3	413C		
		413E		
Grease and Oil	413.1	503A	Sector Sector	1 mg/L
Hardness, Total	130.1	314B		
	130.2	314A		
	215.1+ 242.1			
MBAS	425.1	512A		

(1) Coliform procedures taken from "Microbiological Methods for Monitoring the Environment", 1978, EPA-600/8-78-017.



	- 3eee		· · · .	Page 4.
Analytical Parameter				
	1979 EPA Methods	15th Ed. Standard Methods	rence Others Include Reference & Methods	Min. Report Concentration (incl. Units)
Metals Group I				
Aluminum	202.1	303C		Service and the service
	202.2	304		
	and the second	306B		
Beryllium	210.1	303C		
	210.2	304		
		309B		e
Cadmium	213.1	303A		
and the second	213.2	303B		
and the second		304		
and a second		310B	a har har har har har har har har har ha	
Chromium, Total	218.3	303A		
Uniomium, iotai	218.1	303B		
	218.2	304		
		312A		
Cobalt	219.1	303A	1997 - A. 1997 - A. 1997 - A. 1997 - A.	The start for an
	219.2	303B		and the second
		304		and the second
Copper	220.1	303A		a far and a second
	220.2	303B		Carlo and
	and the second second second	313B		and the second
Iron	236.1	303A		
	236.2	303B	Le contractor and	
		304		
		315B		M Repart Counter Web
Lead	239.1	303A		and the second states
	239.2	303B		a har an an ar
		304		- Frankline
		316B	1	
Manganese	243.1	303A	Children and Chi	-
	243.2	303B		
	A CARLES	304 319B		-

V. 5. Analytical Quality Assurance Procedures

BOD (Biochemical Oxygen Demand)

Normally the BOD samples received by this laboratory are un-Chlorinated. The samples are screened for chlorine before processing. Seeding is therefore, not usually required. The dissolved oxygen meter is calibrated against a Winkler Titration with each batch of samples. Dilution water blanks are set up with each batch of dilution water. When blanks have depletions greater than 0.2 mg/l the corresponding batch of samples are discarded. A set of glucose-glutamic acid standards, with seeding, is set up weekly. Duplications is done daily on one set of samples.

Coliform, Total & Fecal, MF & MPN

Incubator temperatures are read daily. Negative (Pre & Post) and positive controls are run with each set of samples. Any samples but potable water are run with at least two dilutions. Duplicates are also run daily. The laboratory maintains N.C. Microbiology certification for drinking water and all applicable quality control on media, sterilation of equipment, etc.

Oil & Grease

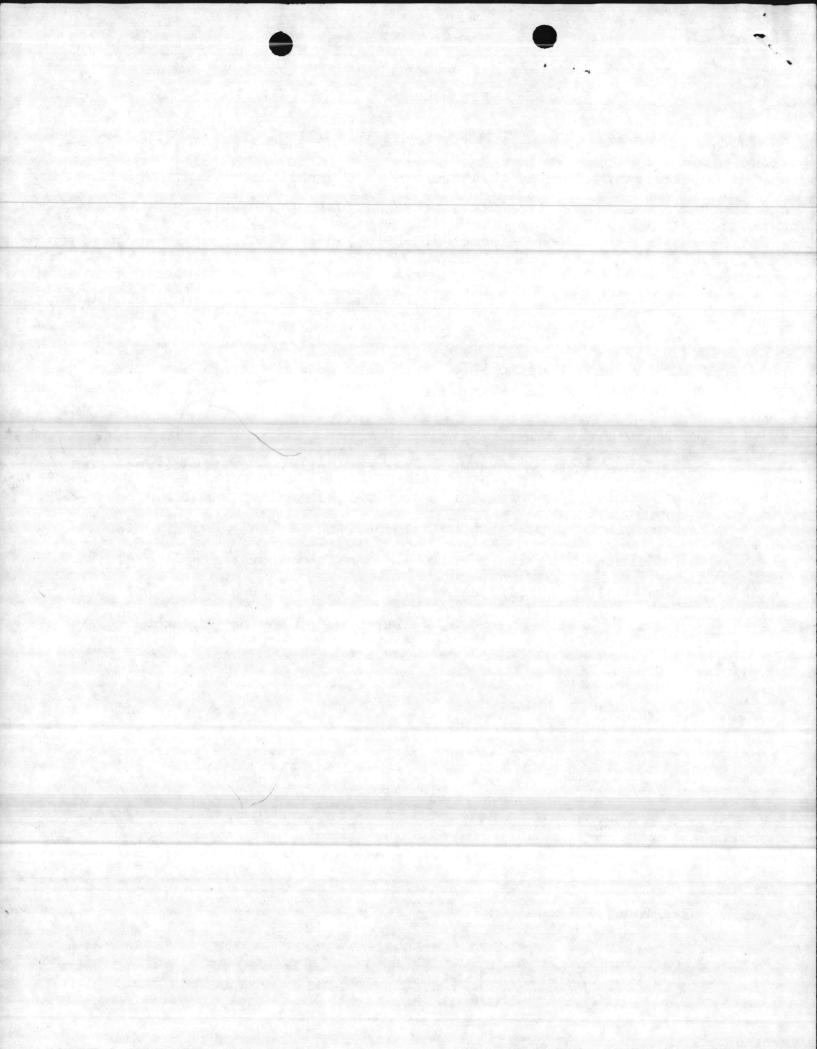
A standard and blank are run with each batch of samples

pH

Meters are standardized using low and high buffers daily. Monthly, meters are checked against certified buffers.

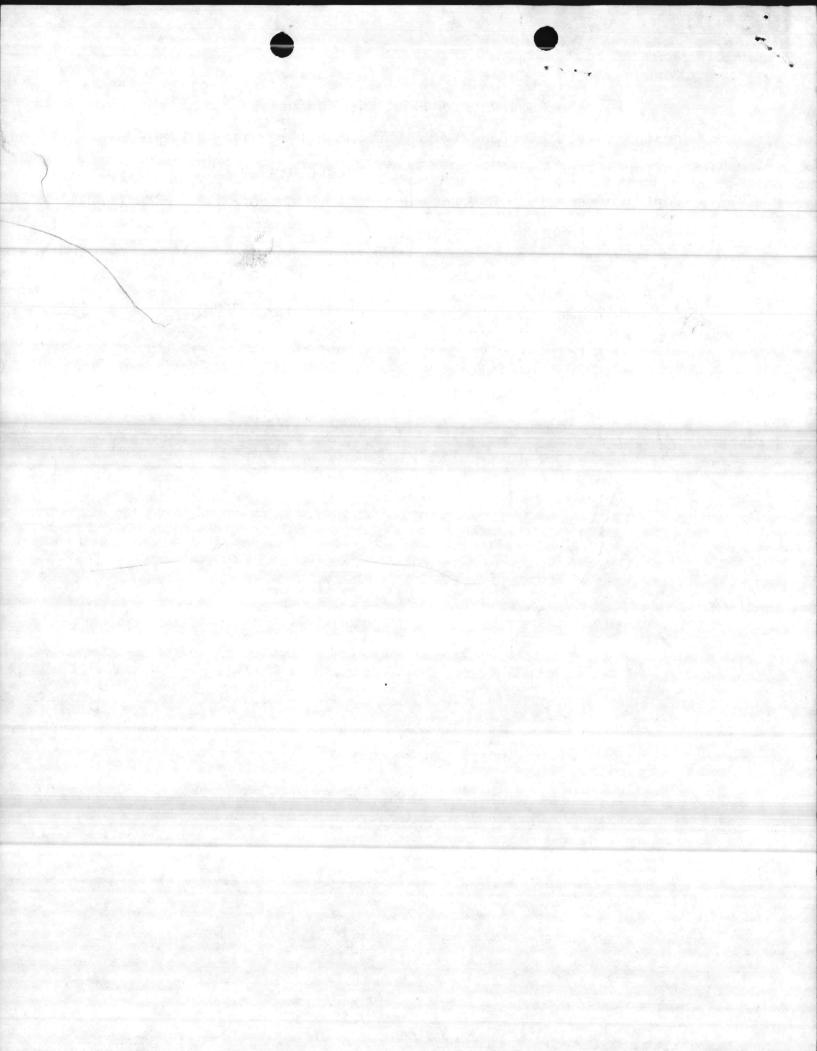
Residue, Solids

Oven temperatures are recorded on Laboratory Worksheets. A duplicate is analyzed daily



General · ·

All balances are zeroed daily. Analytical balance is checked against a set of standard weights monthly. All chemicals are analytical reagent grade. All reagents are dated when prepared. Copies of the 1979 EPA Methods and 15th Ed. of Standard Methods are maintained in the Laboratory.



- B. Other Laboratory Personnel
 - Hoy J. Burns

Education: High School, 1 yr College

Certifications: Dept. of Health, Education & Welfare Clinical Laboratory Technologist, North Carolina Wastewater Operator Grade II

Experience: Navy Clinical Labortories-11 yrs; Navy Research Laboratories-9 yrs; Quality Control Laboratory-9 yrs

Robert J. Lachapeele, Jr. Jachapell,

Education: High School, 2 yrs College, Navy Medical Technologist School

Certifications: Certified Medical Technologist

Experience: Navy Laboratories-17 yrs; Quality Control Laboratory-4 yrs

Gaines B. Huneycutt, Jr

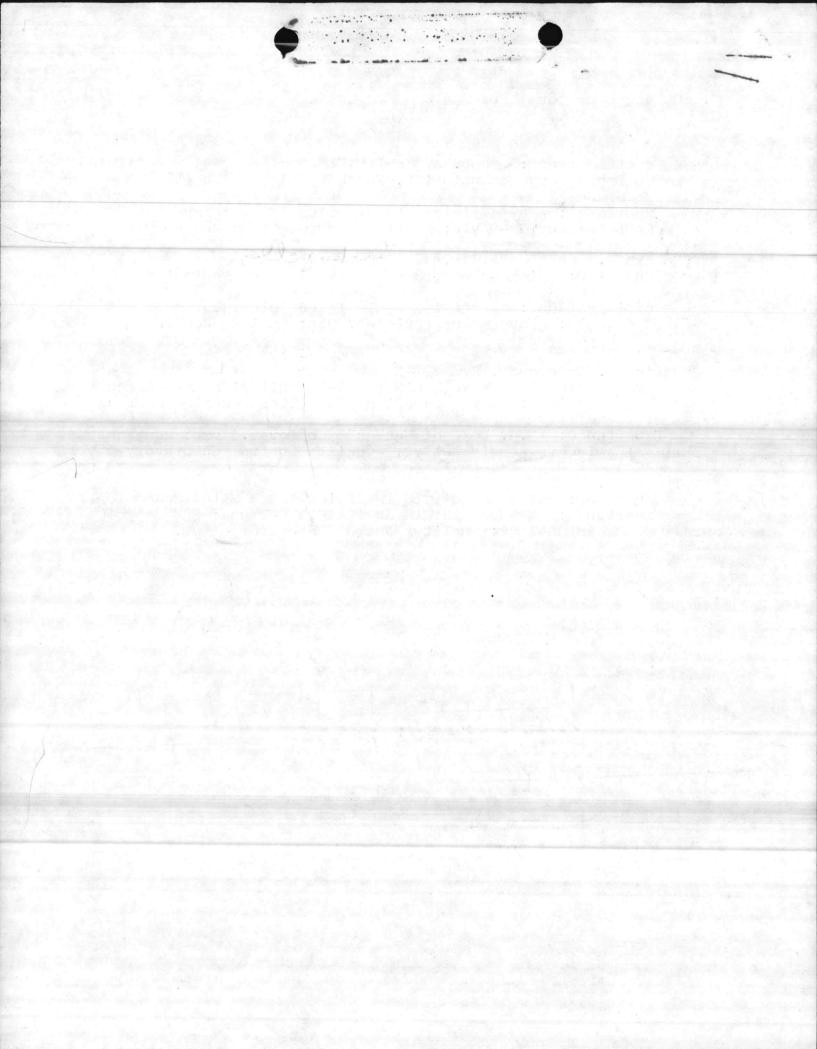
Education: High School, AAS in Fish & Wildlife Management

Certifications: North Carolina Water Treatment Operator Grade C-Well

Experience: N.C. Dept of NatRes & ComDev-6.5 yrs; Contracts & Engineers Services-1 yr; Quality Control Laboratory-4 yrs

Thomas H. Barbee

Education: High School, BS in Biology with Wildlife Option Experience: VA Hospital in Ashville, NC-2 yrs; ECU School of Medicine-1 yr; Quality Control Laboratory-1 yr.



6740/3 NREAD 8 Jul 87

Nr. N. B. Edwards MCRD/DEM Laboratory Branch Post Office Box 27687 Raleigh, North Carolina 27611

12.5

Survey &

Dear Sir:

This is to report that the deviations sited in the 29 April 1987 on-site inspection report have been corrected. The on-site inspection report was received on 11 May 1987. Corrective actions were initiated immediately, and their descriptions are discussed below.

The laboratory now runs the glucose-glutamic acid check for the Bio-chemical Oxygen Demand (BOD) test daily instead of weekly. Two dilutions of each effluent BOD sample is run in addition to the daily duplication of one BOD sample. Duplicates of one sample for Fecal Coliform, Ammonia and Phosphorus are run, in addition to the daily dilutions of each sample.

As stated in our 4 December 1986 letter, the name of the laboratory has changed since our original application in 1985. The laboratory is now the Environmental Chemistry and Microbiology Laboratory.

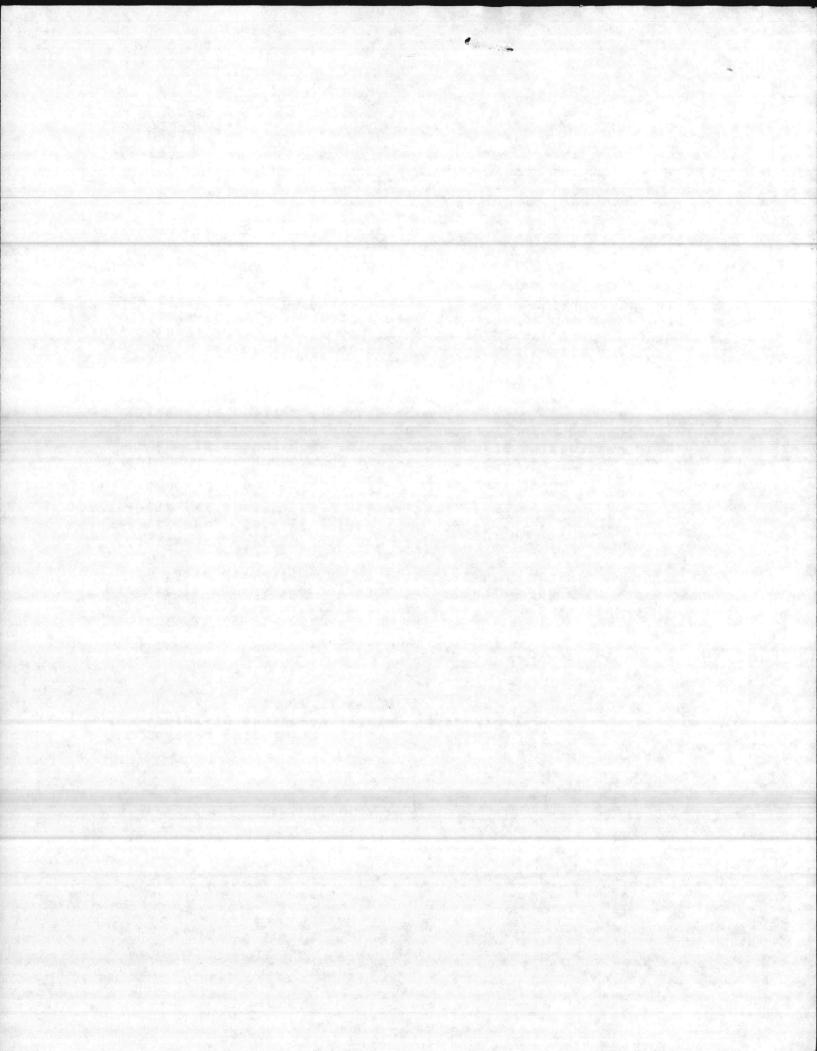
The point of contact on this matter is Ms. Elisabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely.

JULIAN I. WOOTEN Director, Natural Resources Division By direction of the Commanding General

Copy to: ECAML, NREAD (2)

Writer/Typist Betzl Frandi Date Typed & Jul & Word Processor Number (2740/3





State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management

512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary May 7, 1987

R. Paul Wilms Director

Ms. Elizabeth A. Betz Camp Lejeune Water Quality Control Lab Natural Resources & Env Affairs Div, MC Base Camp Lejeune, NC 28542

Dear Ms. Betz:

RE: Initial Laboratory Certification Inspection

Enclosed is a copy of the referenced inspection report as prepared by Mr. Byrd. Each of the deviations must be corrected prior to certification. Within 60 days, please supply this office with a written item for item description of how these deviations were corrected. As a certification requirement, your laboratory must continue to carry out the quality controls set forth in our quality assurance guidance.

Also enclosed for your use is an updated copy of the certification regulation. In this copy, Section .0805(a)(1) has been changed to reference the October 26, 1984 Federal Register.

Thank you for your cooperation during the inspection. Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

W. B. Edwards gr.

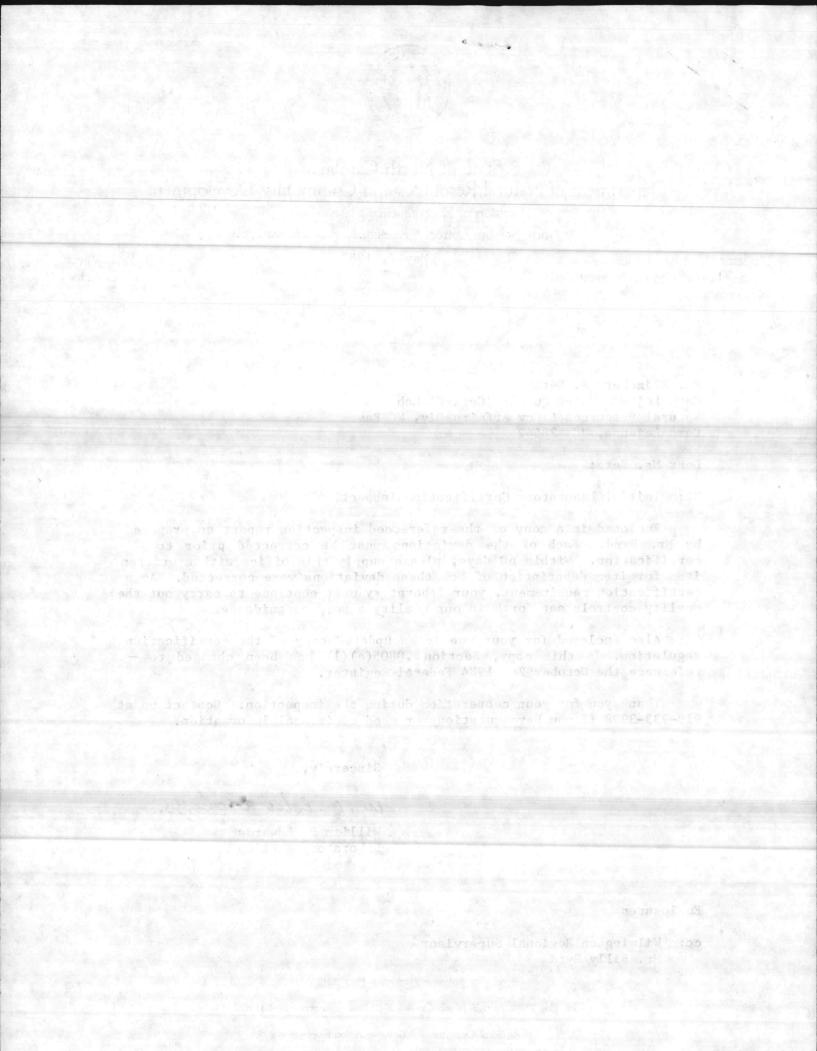
William B. Edwards Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor Mr. Billy Byrd

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



Laboratory Name: Camp Lejeune Water Quality Control Laboratory

Address: Natural Resources & Environmental Affairs Div., MC Base Camp Lejeune, NC 28542

Date of Inspection: April 29, 1987Type: (X) Initial () MaintenanceEvaluator: Billy D. Byrd

Local Person(s) contacted: Ms. Elizabeth A. Betz

- I. Introduction- This laboratory was inspected to verify its compliance with the requirements of NCAC 2H .0800 for the analysis of wastewater samples.
- **II. General Comments:** The laboratory was in good condition including equipment, records, knowledge of tests, quality controls, and personnel.

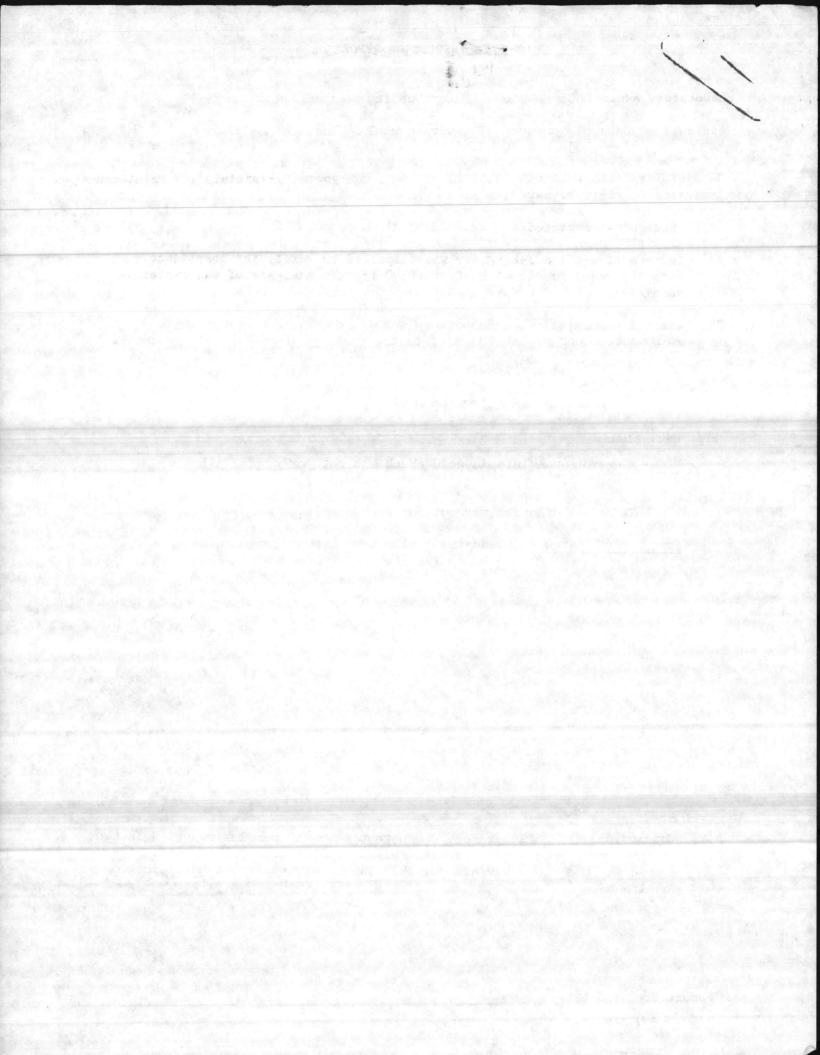
III. Deviations:

BOD - The glucose-glutamic acid check was not performed daily as required.

- Only one dilution was set for each sample.

Sample duplicates - Duplicate samples were not analyzed as required.

IV. Conclusions: This laboratory demonstrated by the results reported in the sample performance evaluation its ability to produce quality data. After correcting the above deviations, this laboratory will meet certification requirements



NRCD/DEM Wastewater Laboratory Certification Guidance for Preparing a Quality Assurance Document

I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

IV. Quality Controls

Listed below are the <u>minimum quality controls required for North Carolina</u> <u>Wastewater Certification</u>. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

⊁ 1. BOD

- a. The temperature of the BOD incubator must be maintained at $20 \pm 1^{\circ}$ C using an accurate thermometer inserted in a BOD bottle inside the incubator.
- b. Check and record the incubator temperature each day.
- c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses.
- d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.

- e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.
- f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/1.
- g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.
- h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.
- i. Analyze a duplicate sample daily.
- j. Perform a blank dilution water control analysis along with each batch of samples analyzed.
- 2. COD Titration Procedure
 - a. Standardize the COD titrant each day samples are analyzed.
 - b. Analyze a distilled water blank with each group of samples and make proper corrections.
 - c. Analyze a quality control standard along with each group of samples analyzed.

Note: A 250 mg/l COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/l COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

- d. Analyze a duplicate sample daily.
- e. Use the low level procedure for the analysis of samples with a COD of <50 mg/1.
- 3. COD Colormetric
 - a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).
 - b. In addition to the calibration standards, analyze a quality control standard each day.
 - c. Analyze a duplicate sample daily.

+4. Coliform

- a. Check the temperature of all incubators daily and maintain a log of values read.
- b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.

- c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.
- d. Log the maximum temperature and pressure of the autoclave once during each use.
- e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.
- f. Analyze one duplicate sample each day.

X 5. Chloride

- a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.
- b. Analyze a distilled water blank each day and make proper corrections.
- c. Analyze one duplicate sample each day.

6. Hardness

- a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.
- b. Analyze a distilled water blank each day samples are analyzed.
- c. Analyze one duplicate sample daily.

× 7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

- a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).
- b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.
- c. Analyze a duplicate sample with each group of samples analyzed.

× 8. Ammonia and Total Kjeldahl Nitrogen - Titration Procedure

- a. Analyze a distilled water blank each day samples are analyzed.
- b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.
- c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.

Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing 1 ml concentrated H_2SO_4 and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated H_2SO_4 to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

4

d. Analyze a duplicate sample daily.

χ 9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

- a. Calibrate the meter according to the manufacturer's instructions.
- b. Check the meter calibration by analyzing a medium level quality control standard each day.
- c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

- a. Calibrate the instrument according to the manufacturer's instructions.
- b. Check the instrument calibration each day by analyzing a low, medium, and high standard.
- c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.
- d. Analyze one duplicate sample each day samples are analyzed.
- e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

a 11. Oil & Grease

- a. Perform a blank analysis on each batch of freon used and make proper corrections.
- b. The freon must be distilled from the extraction flask using a water bath controlled at 70°C.
- c. The extract must be filtered through Whatman #40 filter paper or equivalent.
- d. It is recommended that a reference standard be analyzed quarterly.
- X 12. pH
 - a. Standardize the meter using a low and high buffer daily or before each use.

- b. It is recommended that a reference standard be analyzed quarterly.
- c. Analyze a duplicate sample daily.
- × 13. Total Residue and Total Suspended Residue
 - a. Check and record drying oven temperature each day used.
 - b. Analyze one duplicate sample each day samples are analyzed.
 - c. It is <u>recommended</u> that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.
 - d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

- a. Standards as described in the approved procedure must be secured and used.
- b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.
- c. Analyze one duplicate sample each day samples are analyzed.
- d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.
- 15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- Calibrate the instrument each day as directed in the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily for each parameter.
- e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

- a. Samples must be digested according to the approved procedures.
- b. Set up the instrument according to the manufacturer's instructions.

- c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.
- d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.
- e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

- a. Samples must be digested according to the approved procedures.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.
- c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.
- d. Analyze one duplicate sample daily.

18. Mercury

- a. Set up the instrument according to the manufacturer's instructions and the approved procedure.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.
- c. In addition to the calibration standards, analyze a quality control standard daily.
- d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- b. Set up and calibrate the instrument according to the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily.
- e. Analyze a duplicate sample daily.
- f. Use of the method of standard additions is recommended for all samples.

20. Analytical Balance

- a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.
- b. Weigh a standard weight each day the balance is used and maintain a log of values read.
- c. Check the balance zero before each use.
- d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

- a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.
- b. A copy of the approved reference procedures must be available in the laboratory.

< 22. Chemicals, Reagents, and Glassware

- Reagents must be prepared and used as detailed in the reference procedures.
- b. Date all chemicals received and all reagent solutions prepared.
- c. All chemicals should be reagent grade, when available.
- d. Maintain a record of all standardizations performed.
- e. We recommend that all glassware be Class A, when available.

χ 23. Sample Preservation

- a. Samples should be preserved immediately after collection.
- b. Document the type of preservatives that are to be used and when samples are preserved.

X 24. Records

Analytical and quality control records must be available for inspection and include the following:

- a. Date samples are collected and date analyzed.
- b. Daily lab worksheets and workbooks.
- c. Values obtained on standards, blanks, duplicate samples, and standard curves.

- d. A record of all required quality controls.
- e. All worksheets must contain the signature or initials of the analyst(s) performing that function.
- f. All analytical records must be retained for at least three years.
- 25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed ± 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

a. Precisions Control Limits:

Using 30-40 sets of duplicate sample results or an annual data set. calculate precision control limits using the formulas given below:

Range (R) = 1st analysis - 2nd analysis

$$\overline{R} = \underline{\Sigma R} \\ n$$

$$UWL_R = 2.51 \overline{R}$$

$$UCL_R = 3.27 \overline{R}$$

Where: R

= average range

UWL_p = Upper Warning Limit

- UCL_p = Upper Control Limit
- 2.51 = Shewhart factor for 2s (duplicate)
- 3.27 = Shewhart factor for 3s (duplicate)
- NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1. 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.

b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

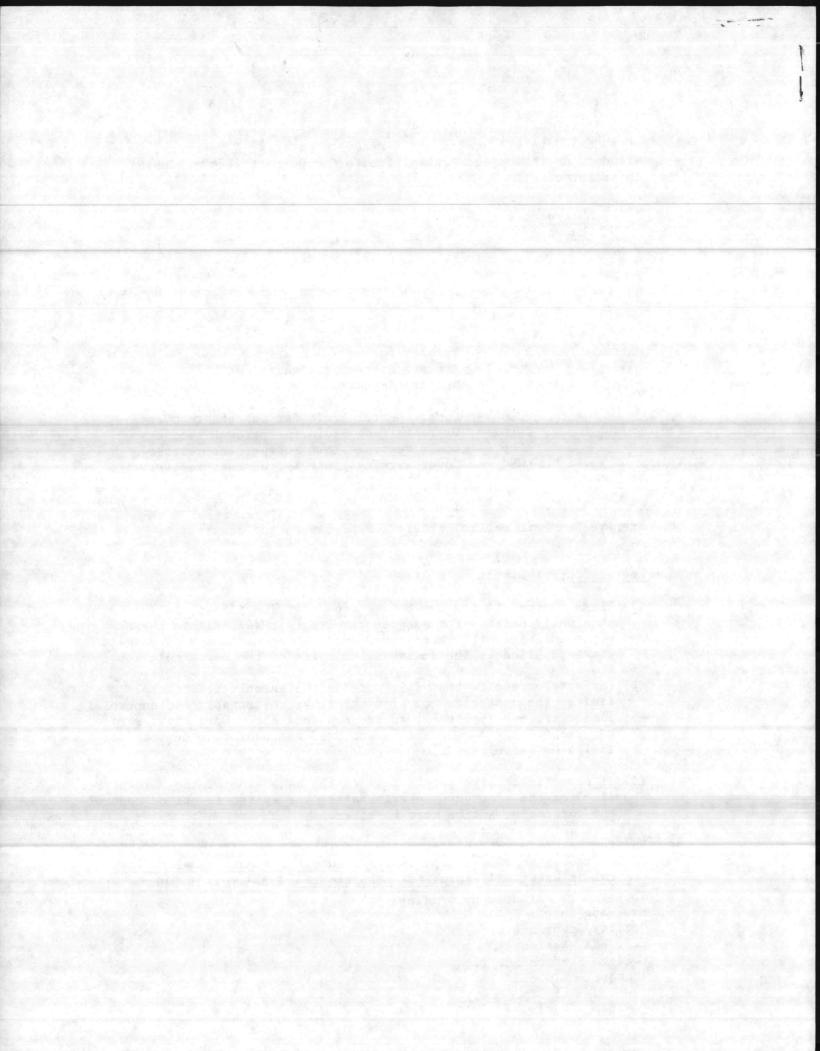
P =	observe known	d x	100								
	KIIOWII				UCL	=	P	+	3	Sp	
D –	50			$\int \Sigma P^2 - (\Sigma P)^2 = \frac{n}{n}$	UWLp	=	P	+	2	Sp	
r =	n			$s_p - \sqrt{\frac{n-1}{n-1}}$	LCLp	=	P	-	3	Sp	
					LWLp	=	P	-	2	Sp	
Where	1	P P		Percent recovery Average percent recovery							
		n	=	Number of analysis							
		Sp	=	Standard deviation of perce	ent ree	ove	ry				

Sp	=	Standard deviation of percent recovery	
UCLp		Upper Control limit for percent recovery	
		Upper warning limit for percent recovery	
LCLp	=	Lower control limit for percent recovery	
LWLp	=	Lower warning limit for percent recovery	

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

- These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.
- (2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.
- (3) If seven successive points fall on the same side of the P (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.
- e. For further information concerning statistical quality control limits, we recommend securing a copy of <u>EPA Handbook for Analytical Quality</u> <u>Control in Water and Wastewater Laboratories</u> EPA-600/4-79-019. This can be obtained by writing: Mr. Wade Knight Quality Assurance Officer U.S. EPA, Region 4 College Station Rd. Athens, GA 30613





R. Paul Wilms

Director

State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management

512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary September 22, 1987

Ms. Elizabeth A. Betz NREAD, EC & M Lab Assistant Chief of Staff, Facilities Camp Lejeune, NC 28542

Dear Ms. Betz:

Re: Initial Wastewater Laboratory Certification

The Department of Natural Resources & Community Development, in accordance with the provisions of N.C. GS 143-215.3(a)(10), 15 NCAC 2H .0800, is pleased to certify your laboratory to perform specified water analysis required by EMC monitoring and reporting regulations 15 NCAC 2B .0500 and 2H .0900.

A certificate acknowledging the certification of your laboratory is enclosed for your use. The certificate describes the requirements and limits of your certification. Please review this certificate to insure that your laboratory is certified for all parameters required to properly meet your certification needs.

Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely. 01.0

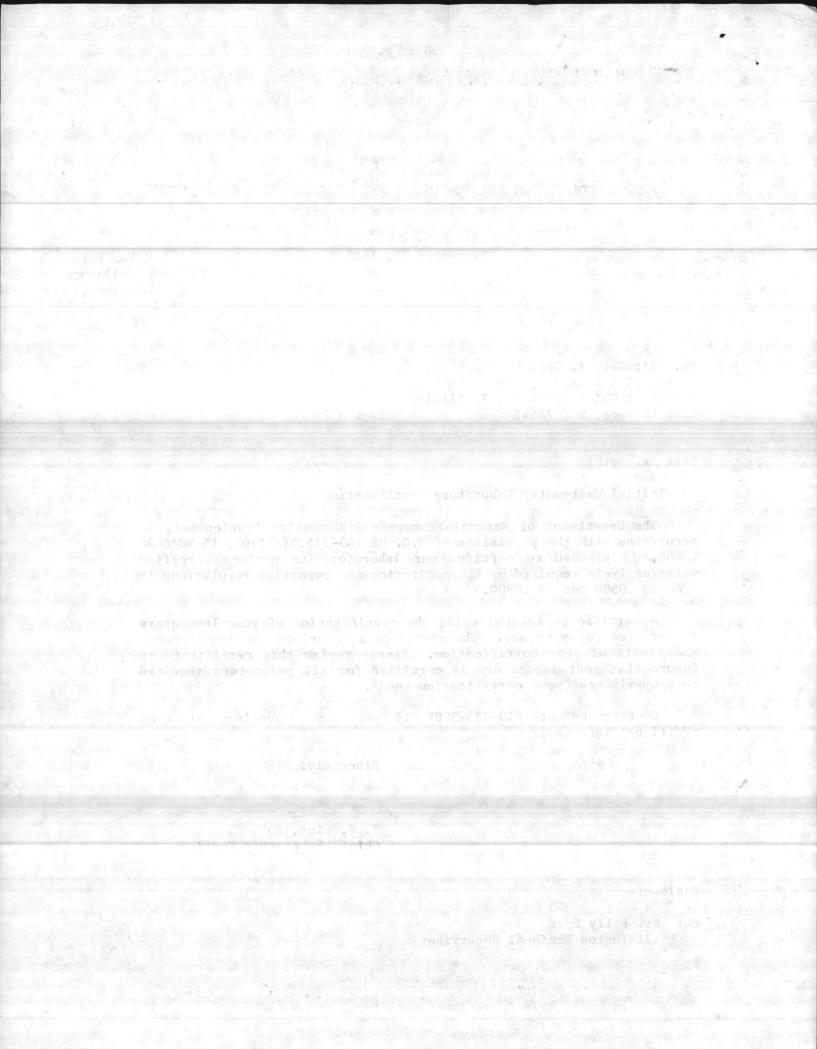
B. E. Sims, PhD Chief, Laboratory Section

Enclosure

cc: Mr. Billy Byrd Wilmington Regional Supervisor

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



STATE OF NORTH CAROLINA DEPARTMENT OF NATURAL RESOURCES & COMMUNITY DEVELOPMENT

Division of Environmental Management Laboratory Certification Program

In accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a) (10) and NCAC 2H .0800:



CAMP LEJEUNE ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LAB

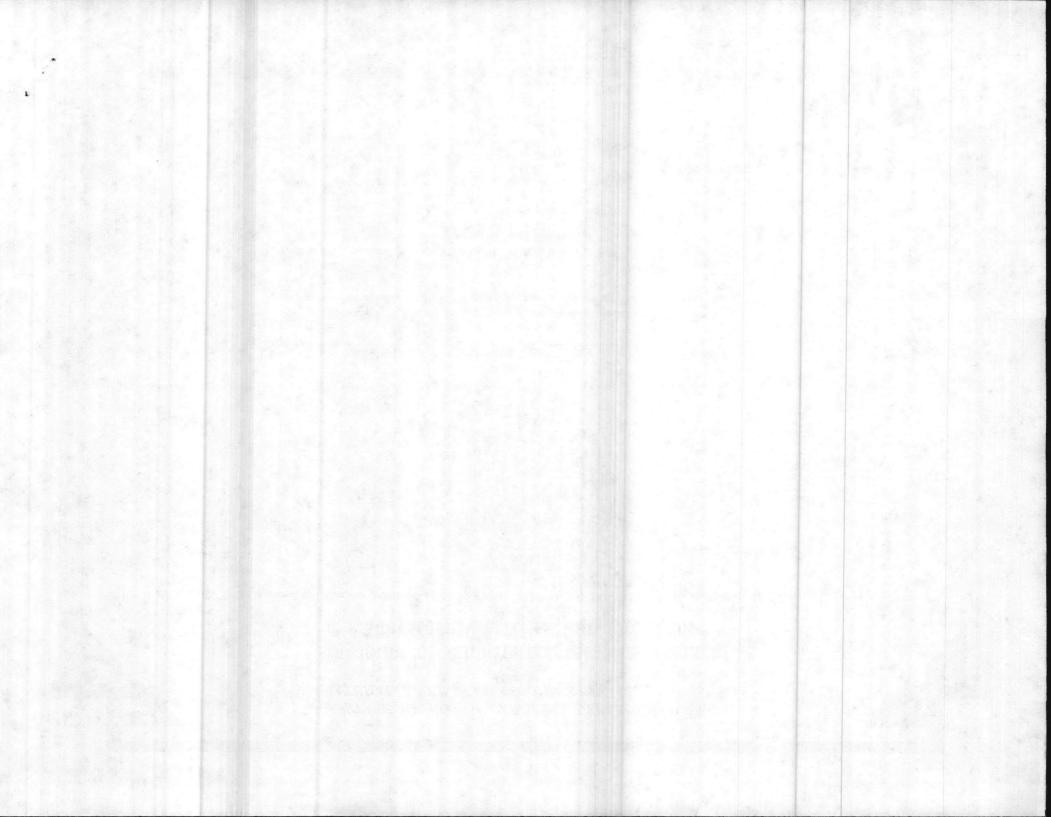
Is hereby certified to perform wastewater analyses (as listed on attachment I) and report monitoring data to DEM for compliance with monitoring and pretreatment regulations.

This certificate does not guarantee validity of data generated, but indicates the methodoloy, equipment, quality control procedures, records, and proficiency of the laboratory have been examined and found to be acceptable.

This certificate shall be valid until _____ December 31, 1989

B. E. Sims, PhD Chief, Laboratory Section

Certificate No. _____227



ATTACHMENT I PARAMETER CERTIFICATION

The Camp Lejeune Environ. Chemistry & Microbiology laboratory has been evaluated and found acceptable for the measurement of the parameters listed below that are preceded by an (X).

X	BOD	Metals, Group I	Arsenic
	COD	aluminum	Barium
	Chloride	beryllium	Mercury
X	Coliform, fecal MF	cadmium	Selenium
X	Coliform, total MF	chromium, total	X Ammonia nitrogen
X	Coliform, fecal tube	cobalt	Total Kjeldahl nitrogen
X	Coliform, total tube	copper	Nitrate + nitrite nitrogen
	Cyanide	iron	X Total phosphorus
-	Fluoride	lead	Orthophosphate
X	Grease and oil	manganese	X pH
	Hardness, total	nickel	Phenols
	MBAS	zinc	Residue, total
		Metals, Group II	X Residue, total suspended
		antimony	Turbidity
, · · · ·		silver	
		thallium	

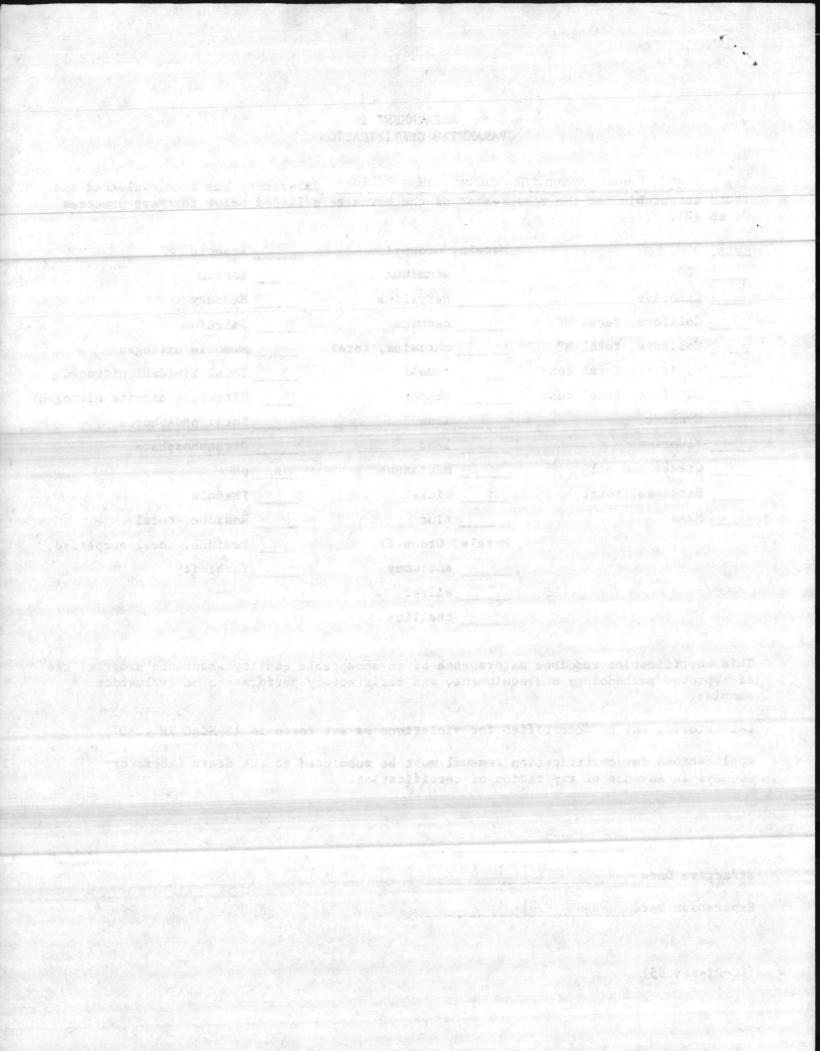
This certification requires maintenance of an acceptable quality assurance program, use of approved methodology and equipment, and satisfactory performance on evaluation samples.

Laboratories may be decertified for violations as set forth in 15 NCAC 2H .0807.

Applications for certification renewal must be submitted to the State Laboratory 30 days in advance of expiration of certification.

Certificate No.	227	
Effective Date	September	21, 1987
Expiration Date	December	31, 1989

(LC-21-Rev.85)





State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management 512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary July 14, 1987

R. Paul Wilms Director

Ms. Elizabeth Betz EC & ML, NREAD Asst. Chief of Staff, Facilities Camp Lejeune, NC 28542

Dear Ms Betz:

Your letter concerning corrective actions for your analytical procedures has been received and reviewed. The actions taken are acceptable and your laboratory meets the requirements for wastewater laboratory certification. Upon receipt of payment for the enclosed invoice, we will issue certification. We have prorated your fees for the remainder of the year. Your laboratory will be subject to a \$250 maintenance fee on January 1, 1988.

Thank you again for your cooperation. Contact us at 919-733-3908 if you have questions.

Sincerely,

W. B. Echvards, Jr.

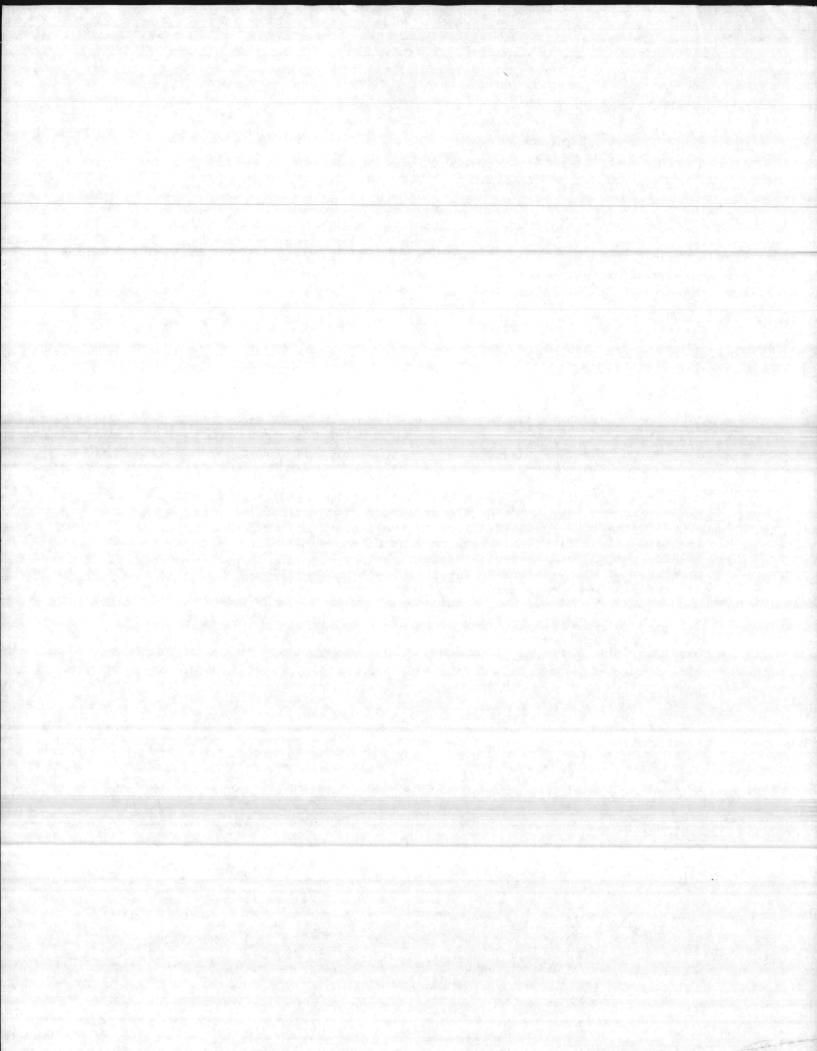
W. B. Edwards Laboratory Section

Enclosures

cc: Mr. Billy Byrd

Pollution Prevention Pays

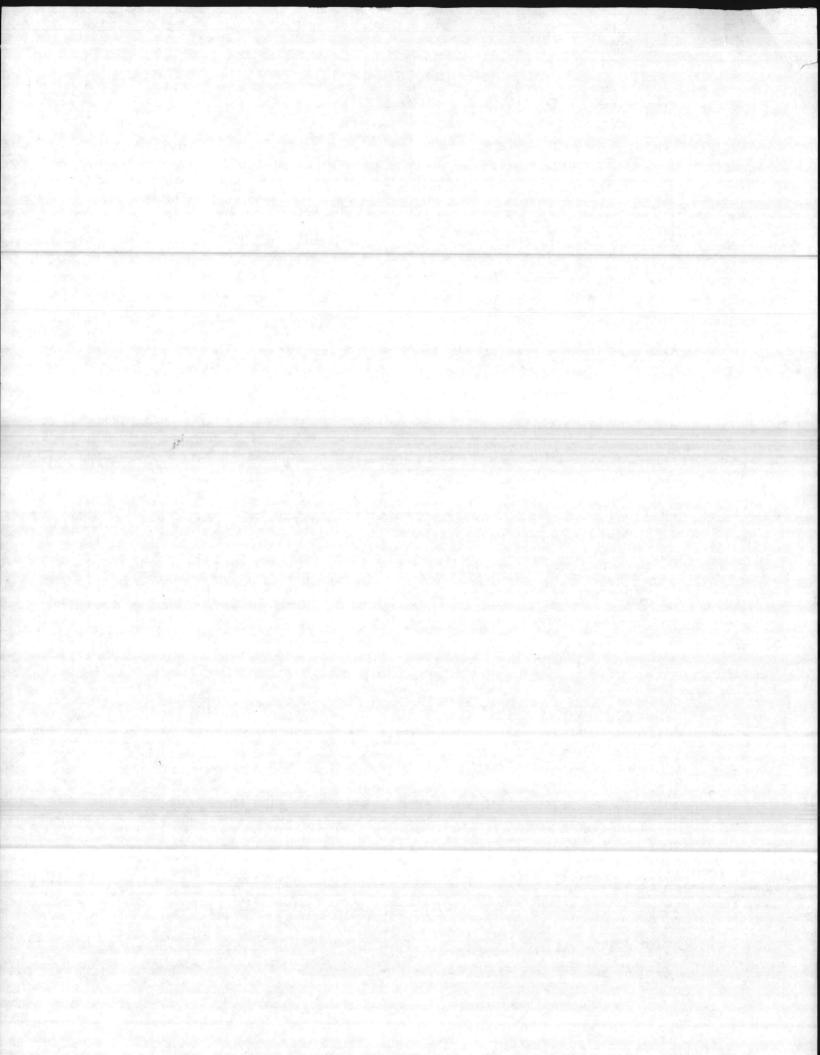
P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



INVOICE

North Carolina Wastewater Laboratory Certification

Laborato	ory Name Camp Le	jeune Environmental	Chem. & Micro L	ab Da	te
Address_	ATTN: Ms. Elizabe	th Betz EC & ML,	NREAD, Asst. Chi	ef of Staff	, Facilities
in the N \$20.00 p an addit	North Carolina Wast per analytical para cional prorated cha		Certification Pr um assessment of te travel expens	ogram. The \$250.00 pe e included	r laboratory with
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	parameters prorat	ted formon	ths:	\$	· · · ·
	Prorated out-of-s	state travel costs:		\$	A second second
			Assessment Due:	\$83.33	
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State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management 512 North Salisbury Street • Raleigh, North Carolina 27611

July 14, 1987

James G. Martin, Governor S. Thomas Rhodes, Secretary R. Paul Wilms Director

Ms. Elizabeth Betz EC & ML, NREAD Asst. Chief of Staff, Facilities Camp Lejeune, NC 28542

Dear Ms Betz:

Your letter concerning corrective actions for your analytical procedures has been received and reviewed. The actions taken are acceptable and your laboratory meets the requirements for wastewater laboratory certification. Upon receipt of payment for the enclosed invoice, we will issue certification. We have prorated your fees for the remainder of the year. Your laboratory will be subject to a \$250 maintenance fee on January 1, 1988.

Thank you again for your cooperation. Contact us at 919-733-3908 if you have questions.

Sincerely,

W. B. Echvards, Jr.

W. B. Edwards Laboratory Section

Enclosures

cc: Mr. Billy Byrd

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015

68. Elisobeth Betz 30 & T. MidaD Asst. Philof of Staff, Facilities Camp I jeune, NO 285(2

Dear Ma Setz:

Your latter a commine corrective act one for your analytical procedures in been realized and reviewed. The actions taken are acceptable and your laboratory meats the requirements for wastewater haboratory certification. Upon receipt of payment for the enclosed invoice, we will issue certification. Ve have promated your fees for the remainder of the year. Four laboratory will be subject to a 250 maintenance fee on January 1 1938.

Thank you as in for your cooperation. Contrat us at 919-723-3908 f you have succtions.

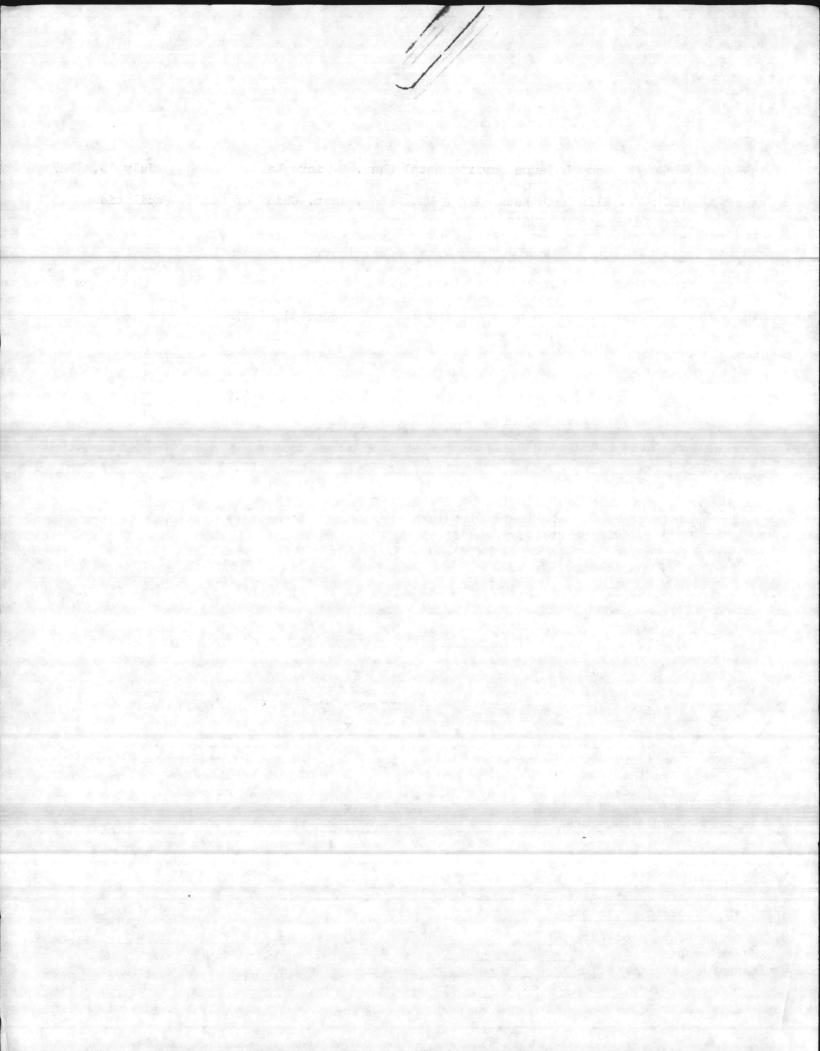
INVOICE

North Carolina Wastewater Laboratory Certification

Auuress_	ATTN: Ms. Elizabeth Betz EC & ML, NREAD, Asst. Ch.	ter of Starr, ractificies
in the N \$20.00 p	Camp Lejeune, NC 28542 lowing statement itemizes the fees required for obtain North Carolina Wastewater Laboratory Certification Proper analytical parameter with a minimum assessment of cional prorated charge for out-of-state travel expension	rogram. The fee schedule is f \$250.00 per laboratory with
The asse	essment for your laboratory is due on <u>August 31</u>	, 1987and includes:
	parameters for one year at \$20 each:	<u>\$</u> .
<13	parameters minimum fee for one year:	\$
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	Prorated out-of-state travel costs:	Ś
		<u>×</u> ¢83_33
	Total Assessment Due	<u>303.33</u> .
	nake your check payable to: <u>NRCD/DEM Lab Certificat</u> ment to: N. C. Department Of Natural Resources & Con DEM Laboratory Section P. O. Box 27687 Raleigh, N. C. 27611 ATTENTION: W. B. Edwards, Jr.	
Mail pay	<pre>/ment to: N. C. Department Of Natural Resources & Con DEM Laboratory Section P. O. Box 27687 Raleigh, N. C. 27611 ATTENTION: W. B. Edwards, Jr. one copy of this invoice with your payment.</pre>	
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State of North Carolina Department of Natural Resources and Community Development Division of Environmental Management 512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor S. Thomas Rhodes, Secretary May 7, 1987

R. Paul Wilms Director

Ms. Elizabeth A. Betz Camp Lejeune Water Quality Control Lab Natural Resources & Env Affairs Div, MC Base Camp Lejeune, NC 28542

Dear Ms. Betz:

RE: Initial Laboratory Certification Inspection

Enclosed is a copy of the referenced inspection report as prepared by Mr. Byrd. Each of the deviations must be corrected prior to certification. Within 60 days, please supply this office with a written item for item description of how these deviations were corrected. As a certification requirement, your laboratory must continue to carry out the quality controls set forth in our quality assurance guidance.

Also enclosed for your use is an updated copy of the certification regulation. In this copy, Section .0805(a)(1) has been changed to reference the October 26, 1984 Federal Register.

Thank you for your cooperation during the inspection. Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

w. B. Edwards gr.

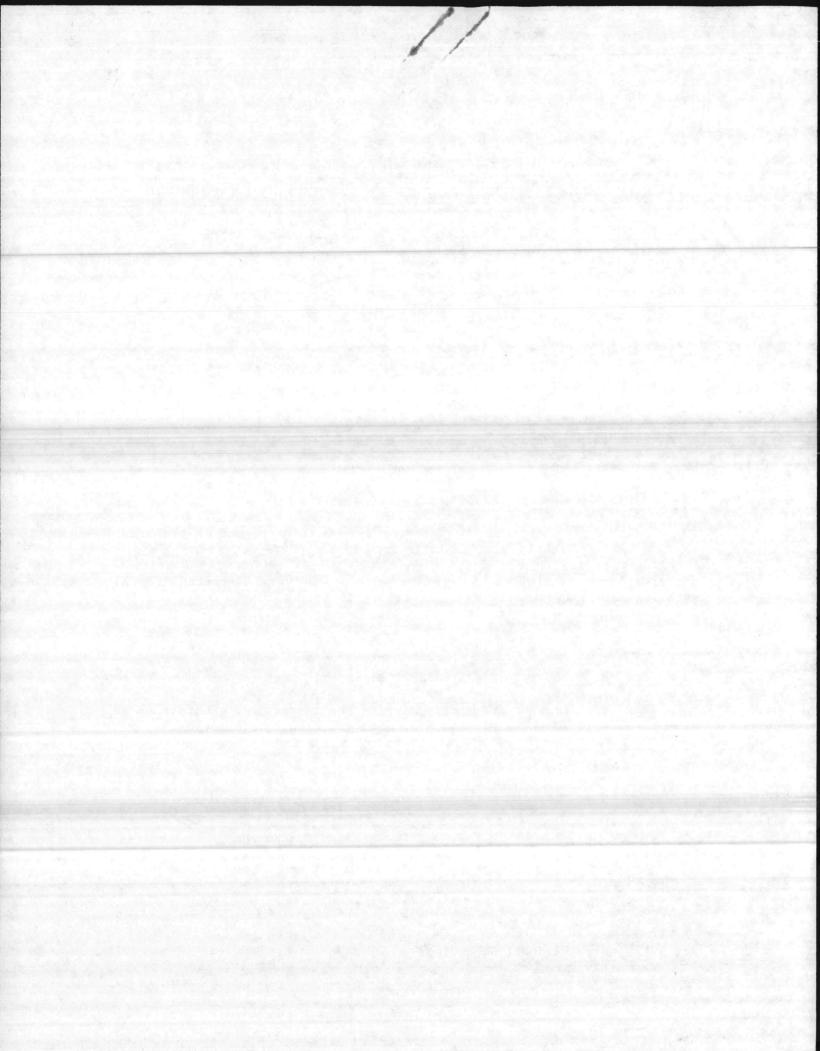
William B. Edwards Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor Mr. Billy Byrd

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015



ON-SITE INSPECTION REPORT

Laboratory Name: Camp Lejeune Water Quality Control Laboratory

Address: Natural Resources & Environmental Affairs Div., MC Base Camp Lejeune, NC 28542

Date of Inspection: April 29, 1987 Evaluator: Billy D. Byrd Type: (X) Initial () Maintenance

Local Person(s) contacted: Ms. Elizabeth A. Betz

- Introduction- This laboratory was inspected to verify its compliance with the requirements of NCAC 2H .0800 for the analysis of wastewater samples.
- II. General Comments: The laboratory was in good condition including equipment, records, knowledge of tests, quality controls, and personnel.

III. Deviations:

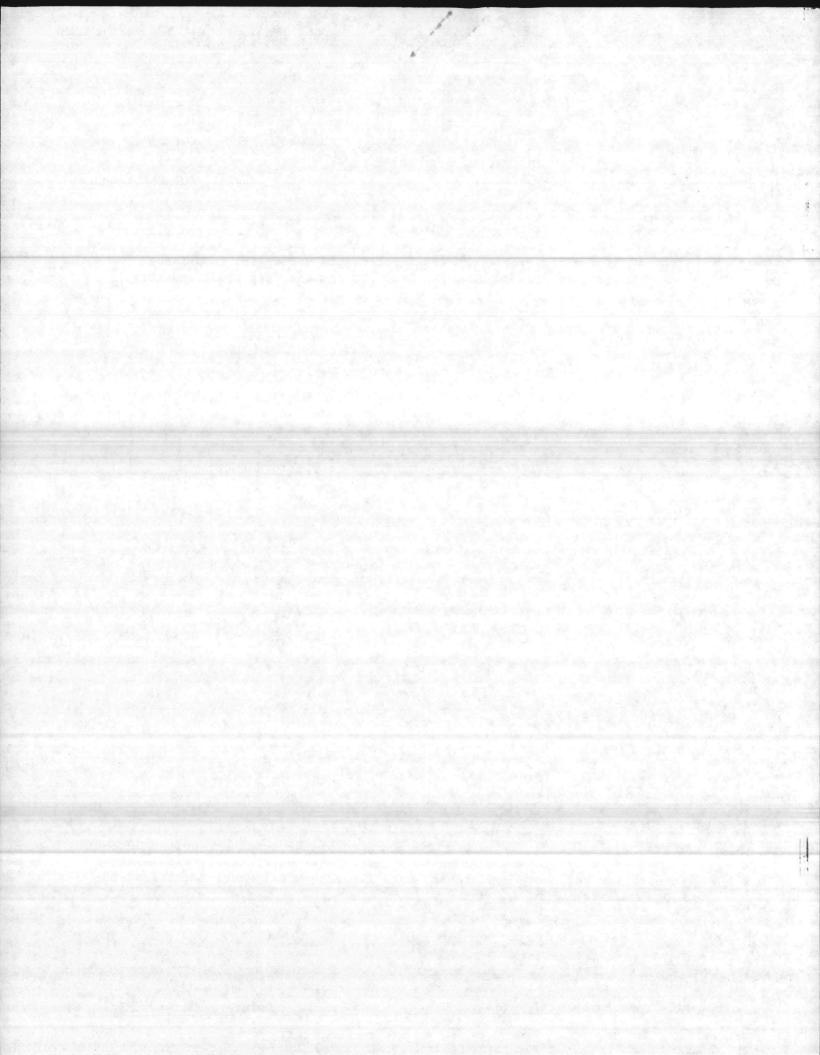
BOD - The glucose-glutamic acid check was not performed daily as required.

- Only one dilution was set for each sample.

Sample duplicates - Duplicate samples were not analyzed as required.

IV. Conclusions: This laboratory demonstrated by the results reported in the sample performance evaluation its ability to produce quality data. After correcting the above deviations, this laboratory will meet certification requirements

Date: May 7, 1987



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TITLE 15

DEPARTMENT OF NATURAL RESOURCES

AND

COMMUNITY DEVELOPMENT

CHAPTER 2

ENVIRONMENTAL MANAGEMENT DIVISION

SUBCHAPTER 2H

PROCEDURES FOR PERMITS, APPROVALS

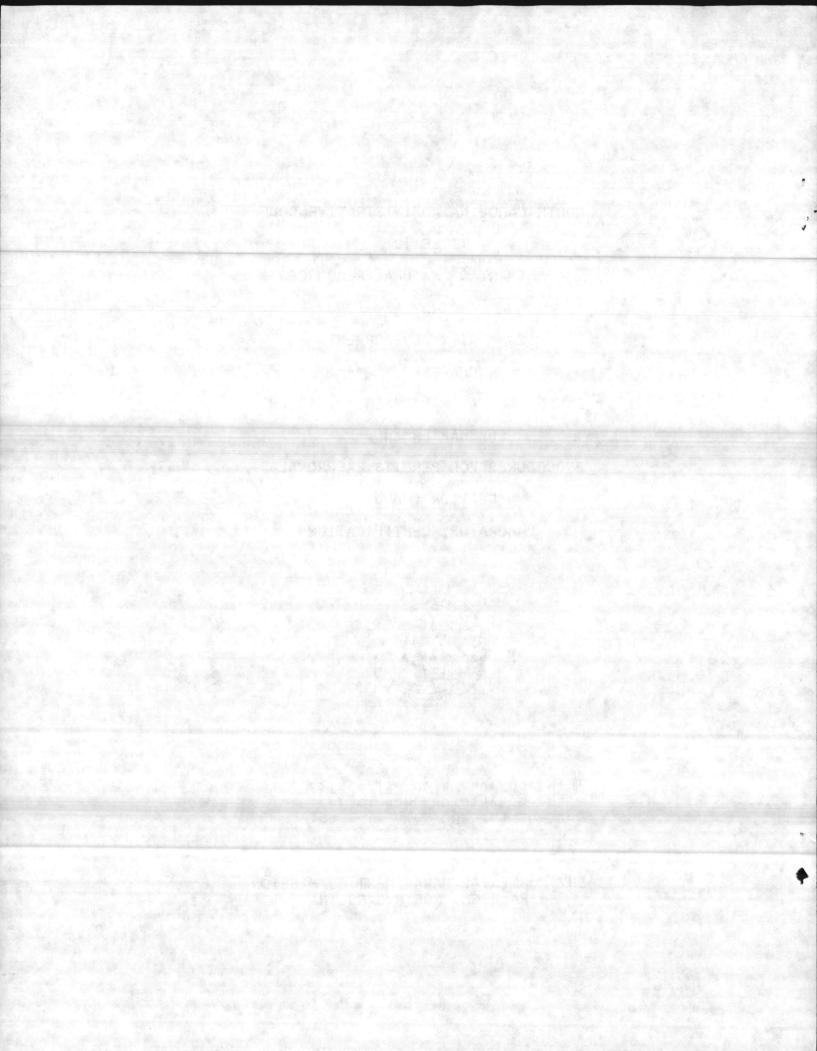
SECTION .0800

LABORATORY CERTIFICATION



This copy has been officially filed with the Attorney General. Current through July 1, 1985.

ENVIRONMENTAL MANAGEMENT COMMISSION RALEIGH, NORTH CAROLINA



SECTION .0800 - LABORATORY CERTIFICATION

.0801 PURPOSE

These Regulations set forth the requirements for state certification of commercial, municipal, and industrial laboratories to perform water analyses, required by the Water and Air Quality Reporting Act, G.S. 143-215.63 et seq; Environmental Management Commission Regulations for Surface Water Monitoring, Reporting, found in Subchapter 2B of this Chapter, Section .0500 and Environmental Management Commission Regulations for Local Pretreatment Programs, found in 15 NCAC 2H .0900.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0802 SCOPE

These Regulations apply to commercial laboratories and Class III and IV municipal or industrial wastewater treatment plant laboratories which perform water analyses for persons subject to G.S. 143-215.1, 143-215.63, et seq., or the Environmental Management Commission Regulations for Surface Water Monitoring, Reporting found in Subchapter 2B of this Chapter, Section .0500. These Regulations also apply to all wastewater treatment plant laboratories for municipalities having Local Pretreatment Programs as found in 15 NCAC 2H .0900. Municipal and industrial laboratories that perform analyses for two or less of the parameters listed in Paragraph .0804(a) of these Regulations are exempt from the requirements of these Regulations.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0803 DEFINITIONS

The following terms as used in this Section shall have the assigned meaning:

- (1) Commercial Laboratory means any laboratory which is seeking to analyze water samples for others.
- (2) State means the North Carolina Division of Environmental Management of the Department of Natural Resources and Community Development, or its successor.

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- (3) State Laboratory means the laboratory branch of the North Carolina Division of Environmental Management, or its successor.
- (4) Unacceptable results on performance evaluation samples or split samples are those that vary by more than plus or minus 25 percent of the value determined by the State Laboratory or the State Laboratory may adopt specific variance limits for a particular parameter.
- (5) Certification is a declaration by the state that the personnel, equipment, records, quality control procedures, and methodology cited by the applicant are accurate and that the applicant's proficiency has been considered and found to be acceptable.
- (6) Decertification is loss of certification.
- (7) Recertification is reaffirmation of certification.
- (8) Municipal Laboratory means a laboratory operated by a municipality or other local government to analyze samples from its wastewater treatment plant(s).
- (9) Industrial Laboratory means a laboratory operated by an industry to analyze samples from its wastewater treatment plant(s).
- (10) Pretreatment Program means a program of waste pretreatment requirements set up in accordance with 15 NCAC 2H .0900 and approved by the Division of Environmental Management.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0804 PARAMETERS FOR WHICH CERTIFICATION MAY BE REQUESTED

Commercial laboratories need to obtain certification only for parameters which will be reported by the client to comply with the monitoring and pretreatment regulations. Municipal and Industrial Laboratories need to obtain certification only for parameters which will be reported to the state to comply with monitoring and pretreatment regulations. A listing of selected parameters follows:

- (1) BOD
- (2) COD
- (3) Chloride
- (4) Coliform, fecal MF
- (5) Coliform, total MF
- (6) Coliform, fecal tube
- (7) Coliform, total tube
- (8) Cyanide
- (9) Fluoride

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- Grease and Oil (10)
- (11) Hardness, total
- (12) MBAS
- (13) Metals, Group I
 - (a) aluminum
 - (b) bervllium
 - (c) cadmium
 - (d) chromium, total
 - (e) cobalt

 - (f) copper (g) iron (h) lead
 - (i) maganese
 - (j) nickel
 (k) zinc
- (14) Metals, Group II
 - (a) antimony
 (b) silver

 - (c) thallium
- (15) Arsenic
- (16) Barium
- (17) Mercury
- (18) Selenium
- (19) Ammonia nitrogen
- (20) Total Kjeldahl nitrogen (TKN)
- (21) Nitrate plus nitrite nitrogen
- (22) Total phosphorus
- (23) Orthophosphate
- (24)pH
- (25) Phenols
- (26) Residue, total
- Residue, total suspended (27)
- (28) Turbidity

History Note: Statutory Authority G.S. 143-215.3(a) (1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0805 CERTIFICATION AND RENEWAL OF CERTIFICATION

(a) Prerequisites for Certification

Laboratory Procedures. Analytical methods, sample preservation, sample containers and sample holding (1) times shall conform to those requirements found in 40 CFR-136.3, Federal Register, Vol. 49, p. 43234 (October 26, 1984); and, Federal Register, Vol. 50, p. 690

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(January 4, 1985), as submitted by the Environmental Protection Agency.

- (2) Performance Evaluations. Each laboratory must demonstrate satisfactory performance on evaluation samples submitted by the State Laboratory. (3)
 - Supervisory Requirements.
 - The supervisor of a commercial laboratory must (A) have a minimum of a B.S. or A.B. degree from an accredited college or university in chemistry or closely related science curriculum plus a minimum of two years laboratory experience in analytical chemistry, or a two year associate degree from an accredited college, university, or technical institute in chemistry technology, environmental sciences, or closely related science curriculum plus a minimum of four years experience in analytical chemistry. Non-degree supervisors must in have at least six years laboratory experience.
 - The supervisor of a municipal or industrial waste (B) water treatment plant laboratory must have a a B.S. or A.B. degree from an minimum of accredited college or university in chemistry or closely related science curriculum plus a minimum of six months laboratory experience in analytical chemistry, or a two year associate degree from an accredited college, university, or technical institute in chemistry technology, environmental sciences, or closely related science curriculum plus a minimum of two years experience in analytical chemistry. Non-degree supervisors must have at least six years laboratory experience.
 - All laboratory supervisors are subject to review (C) by the State Laboratory. One person may serve as supervisor of no more than two laboratories. The supervisor shall provide personal and direct supervision of the technical personnel and be held responsible for the proper performance and reporting of all analysis made for these If the supervisor is to be absent, Regulations. the supervisor shall arrange for a substitute capable of insuring the proper performance of all laboratory procedures. Existing laboratory supervisors that do not meet the requirements in this Paragraph may be accepted after review by the State Laboratory and meeting all other certification requirements.

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- (4) Application. Each laboratory requesting state certification or certification renewal shall submit an application in duplicate to the State Laboratory. Each application will be reviewed to determine the adequacy personnel, equipment, records, quality control of procedures, and methodology. After receiving a completed application and prior to issuing certification, a representative of the State Laboratory may visit each laboratory to verify the information in the application and the adequacy of the laboratory.
- (5) Facilities and equipment. Each laboratory requesting certification must contain or be equipped with the following:
 - (A) A minimum of 150 sq ft of laboratory space;
 - (B) A minimum of 12 linear feet of laboratory bench space;
 - (C) A sink with hot and cold water;
 - (D) Adequate lighting, cooling, and heating;
 - (E) An analytical balance capable of weighing 0.1 mg, mounted on a heavy shock proof table;
 - (F) A refrigerator of adequate size that will maintain temperature of 4°C;
 - (G) An EPA approved or a current copy of "Standard Methods for the Analysis of Water and Wastewater" or EPAs "Methods for Chemical Analysis of Water and Wastes";
 - (H) A source of distilled or deionized water that will meet the minimum criteria of the approved methodologies;
 - Glassware, chemicals, supplies, and equipment required to perform all analytical procedures included in their certification.
- (6) Analytical Quality Control Program. Each laboratory shall develop and maintain a document outlining the analytical quality control practices used for the parameters included in their certification. Supporting records shall be maintained as evidence that these practices are being effectively carried out. The quality control program shall be available for inspection by the State Laboratory and include the following:
 - (A) All analytical quality control data pertinent to each certified analysis must be available for inspection upon request.
 - (B) Analyze one duplicate sample and one known standard in addition to calibration standards each

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day samples are analyzed to document precision and accuracy.

- (C) Any quality control procedures required by a particular approved method shall be considered as required for certification for that analysis.
- (D) All quality control requirements as set forth by the State Laboratory.
- (E) A corrective action policy requiring that at any time quality control results indicate an analytical problem, resolve the problem and rerun any samples involved.
- (F) A policy requiring that all analytical records must be maintained for a period of three years.
- (b) Issuance of Certification
 - (1) In the absence of substantial deficiencies, certification will be issued by the Director, Division of Environmental Management, Department of Natural Resources and Community Development, or his delegate, for each of the applicable parameters requested.
 - (2) Initial certifications will be issued for prorated time periods to schedule all certification renewals on the first day of January.
 - (3) Initial certification shall be valid for up to three years from date of issue.
- (c) Maintenance of Certification.
 - (1) To maintain certification for each parameter, a certified laboratory must analyze up to three performance evaluation samples per year submitted by the State Laboratory as an unknown. Laboratories submitting unacceptable results on a performance evaluation sample may be required to analyze more than three samples per year.
 - (2) In addition, the State Laboratory may request that samples be split into two equal representative portions, one part going to the state and the other to the certified laboratory for analysis.
 - (3) A certified laboratory will be subject to periodic inspections during the certification period and shall make time and records available for inspections.
 - (4) The State Laboratory will maintain a list of certified commercial laboratories and the parameters for which the laboratories have been certified. The list will be revised every six months.
 - (5) The State Laboratory will maintain a list of certified municipal and industrial laboratories and the parameters for which the laboratories have been certified. The list will be revised every six months.

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- (6) A certified laboratory must provide the State Laboratory with written notice of laboratory supervisor changes within 30 days of such changes.
- (d) Certification Renewals.
 - Applications for certification renewal will be submitted in duplicate to the State Laboratory 30 days in advance of expiration of certification.
 - (2) Certification renewals of laboratories shall be issued for three years with the exception that renewals for existing certified laboratories may be prorated to make all certification renewals due on the first day of January.
- (e) Discontinuation of Certification
 - A laboratory may discontinue certification for any or all parameters by making a written request to the State Laboratory.
 - (2) After discontinuation of certification, a laboratory may be recertified by meeting the requirements for initial certification.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. July 1, 1985; December 1, 1984; November 1, 1978.

.0806 FEES ASSOCIATED WITH CERTIFICATION PROGRAM

(a) Certification and Certification Renewal Fees. Before being granted certification or certification renewal, laboratories shall pay to the state a fee of twenty dollars (\$20.00) for each parameter for which certification is requested, however, the minimum fee will be two hundred fifty dollars (\$250.00).

(b) Certification Maintenance Fees. After certification or certification renewal has been issued certified laboratories will pay to the state a certification maintenance fee of two hundred fifty dollars (\$250.00) each year. Certification maintenance fees will not be required for those years in which certification or certification renewal are required. These fees are due on or before the first day of January or the certification anniversary date.

(c) Fees may be prorated in order to make all certification renewals due on the first day of January.

(d) Out-of-state laboratories shall reimburse the state for actual travel and subsistence costs incurred in certification and maintenance of certification.

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History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0807 DECERTIFICATION

(a) Laboratory Decertification. Once certified, a laboratory

- may lose its certification for all parameters by failing to:
 (1) Maintain the facilities, or records, or personnel, or equipment, or quality control program as set forth in the application and these Regulations; or
 - (2) Submit truthful and accurate data reports; or
 - (3) Pay required fees by the date due; or
 - (4) Discontinue supplying data for clients or programs described in Rule .0802 of this Section during periods when a parameter decertification is in effect.

(b) Parameter Decertification. Once certified, a laboratory may loose its certification for a a parameter by failing to:

- Obtain acceptable results on two consecutive performance evaluation samples submitted by the State Laboratory; or
- (2) Obtain acceptable results on three consecutive split samples that have also been analyzed by the State Laboratory; or
- (3) Submit a split sample to the State Laboratory as requested; or
- (4) Use approved methods of analysis; or
- (5) Report equipment changes that would affect its ability to perform the test within 30 days of such changes; or
- (6) Report analysis of performance evaluation samples submitted by the State Laboratory within 30 days of receipt; or
- (7) Maintain records and perform quality controls as set forth by these Regulations and the State Laboratory for a particular parameter; or

(8) Maintain equipment required for a particular parameter.

(c) Decertification Requirements.

- A decertified laboratory is not to analyze samples for the decertified parameters for programs described in Rule .0802 of this Section or clients reporting to these programs.
- (2) A decertified commercial laboratory must make arrangements to supply analysis through a certified laboratory during any decertification periods or notify clients that the analysis cannot be supplied. The decertified laboratory must supply the State Laboratory

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with the name of the laboratory to be used and the client(s) involved.

(3) A decertified municipal or industrial laboratory must make arrangements to have their samples analyzed by another certified laboratory during any decertification period and supply the State Laboratory with the name of the certified laboratory to be used.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0808 RECERTIFICATION

(a) A laboratory decertified because of failure to maintain sufficient or adequate facilities, or laboratory supervisor, or records, or equipment, or quality control program, or failure to pay required fees may be recertified after 30 days by showing to the satisfaction of the State Laboratory that it has corrected the deficiency(ies).

(b) A laboratory decertified for a parameter due to unacceptable results on two consecutive performance evaluation samples submitted by the State Laboratory, or on three consecutive split samples may be recertified after 60 days by reporting acceptable results on two performance evaluation samples similar to those for which approval was lost. Recertification samples may be requested at any time, however, recertification must be requested in writing at the end of the 60 day period immediately following the date of decertification.

(c) A laboratory decertified for falsified reports looses certification for all parameters and shall not be considered for any certification for a one-year period.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0809 RECIPROCITY

(a) Laboratories certified under other state certification programs may be given reciprocity certification where such programs meet the requirements of these Regulations. In requesting reciprocity certification, laboratories shall include with the application required by Regulation .0805(a) of this Section a copy of their certification and Regulation from the certifying agency.

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(b) Laboratories certified on the basis of program equivalency shall pay the fees required by Regulation .0806 of this Section.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0810 ADMINISTRATION

(a) The Director of the Division of Environmental Management, Department of Natural Resources and Community Development, or his delegate, is authorized to issue certification, to reject applications for certification, to renew certification, to issue recertification, to issue decertification, and to issue reciprocity certification.

(b) Appeals. In any case where the Director of the Division of Environmental Management, Department of Natural Resources and Community Development or his delegate denies certification, or decertifies a laboratory, the laboratory may appeal to the director or his delegate for a hearing. Upon receipt of such a request, the director or his delegate shall convene a hearing of the type provided for Environmental Management Commission Regulation in 15 NCAC 2I .0300, Administrative Hearings. Appeal from the decision of a hearing officer appointed by the director or his delegate shall be governed by the Environmental Management Commission Regulation on administrative hearings.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0811 IMPLEMENTATION

- (a) Commercial Laboratories
 - Certified commercial laboratories must meet any new requirements set forth herein within 6 months of these Regulations effective date.
 - (2) Certification fee changes are effective January 1, 1985.
 - (3) Requests for new parameters can be made by submitting a proper application form.
- (b) Municipal and Industrial Laboratories
 - All Municipal and Industrial Waste Treatment Plant Laboratories subject to Rule .0802 of these Regulations are required to be certified.
 - (2) All Municipal and Industrial Waste Treatment Plant Laboratories subject to Rule .0802 of these Regulations

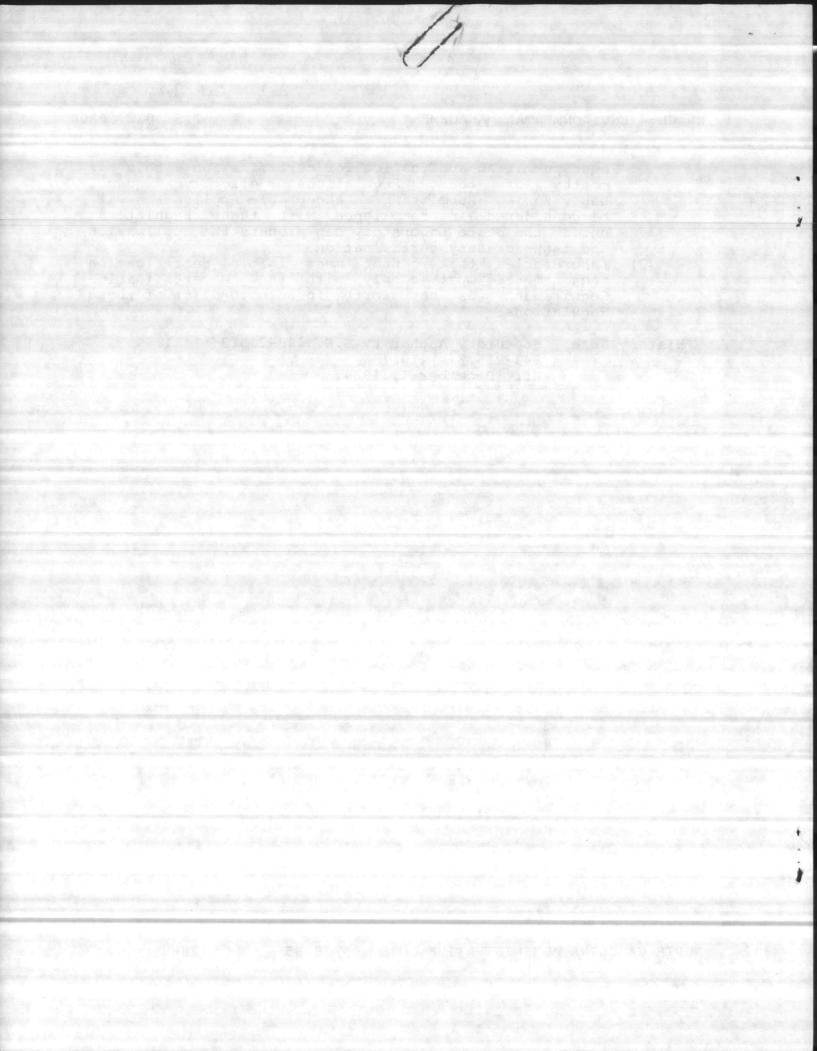
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must submit an application for certification within six months of these Regulations effective date. Laboratories submitting an acceptable application will be considered in compliance with these Regulations until the State Laboratory can process the application and issue or deny certification.

(3) Laboratories that cannot meet initial certification requirements must comply with the Decertification Requirements as set forth in Rule .0807(c) of these Regulations.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. December 1, 1984.

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NRCD/DEM Wastewater Laboratory Certification Guidance for Preparing a Quality Assurance Document

I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

IV. Quality Controls

Listed below are the minimum quality controls required for North Carolina Wastewater Certification. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

1. BOD

- a. The temperature of the BOD incubator must be maintained at 20 ± 1°C using an accurate thermometer inserted in a BOD bottle inside the incubator.
- b. Check and record the incubator temperature each day.
- c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses.
- d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.

- e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.
- f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/1.
- g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.
- h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.
- i. Analyze a duplicate sample daily.
- j. Perform a blank dilution water control analysis along with each batch of samples analyzed.
- 2. COD Titration Procedure
 - a. Standardize the COD titrant each day samples are analyzed.
 - b. Analyze a distilled water blank with each group of samples and make proper corrections.
 - c. Analyze a quality control standard along with each group of samples analyzed.

Note: A 250 mg/1 COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/1 COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

- d. Analyze a duplicate sample daily.
- e. Use the low level procedure for the analysis of samples with a COD of <50 mg/l.

3. COD - Colormetric

- a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).
- b. In addition to the calibration standards, analyze a quality control standard each day.
- c. Analyze a duplicate sample daily.

4. Coliform

- a. Check the temperature of all incubators daily and maintain a log of values read.
- b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.

- c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.
- d. Log the maximum temperature and pressure of the autoclave once during each use.
- e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.
- f. Analyze one duplicate sample each day.

5. Chloride

- a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.
- b. Analyze a distilled water blank each day and make proper corrections.
- c. Analyze one duplicate sample each day.

6. Hardness

- a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.
- b. Analyze a distilled water blank each day samples are analyzed.
- c. Analyze one duplicate sample daily.

7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

- a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).
- b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.
- c. Analyze a duplicate sample with each group of samples analyzed.
- 8. Ammonia and Total Kjeldahl Nitrogen Titration Procedure
 - a. Analyze a distilled water blank each day samples are analyzed.
 - b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.
 - c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.

Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing 1 ml concentrated H_2SO_4 and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated H_2SO_4 to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

d. Analyze a duplicate sample daily.

9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

- a. Calibrate the meter according to the manufacturer's instructions.
- b. Check the meter calibration by analyzing a medium level quality control standard each day.
- c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

- a. Calibrate the instrument according to the manufacturer's instructions.
- b. Check the instrument calibration each day by analyzing a low, medium, and high standard.
- c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.
- d. Analyze one duplicate sample each day samples are analyzed.
- e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

11. Oil & Grease

- a. Perform a blank analysis on each batch of freon used and make proper corrections.
- b. The freon must be distilled from the extraction flask using a water bath controlled at 70° C.
- c. The extract must be filtered through Whatman #40 filter paper or equivalent.
- d. It is recommended that a reference standard be analyzed quarterly.
- 12. pH
 - a. Standardize the meter using a low and high buffer daily or before each use.

- b. It is recommended that a reference standard be analyzed quarterly.
- c. Analyze a duplicate sample daily.

13. Total Residue and Total Suspended Residue

- a. Check and record drying oven temperature each day used.
- b. Analyze one duplicate sample each day samples are analyzed.
- c. It is recommended that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.
- d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

- a. Standards as described in the approved procedure must be secured and used.
- b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.
- c. Analyze one duplicate sample each day samples are analyzed.
- d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- b. Calibrate the instrument each day as directed in the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily for each parameter.
- e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

- a. Samples must be digested according to the approved procedures.
- b. Set up the instrument according to the manufacturer's instructions.

- c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.
- d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.
- e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

- a. Samples must be digested according to the approved procedures.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.
- c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.
- d. Analyze one duplicate sample daily.

18. Mercury

- a. Set up the instrument according to the manufacturer's instructions and the approved procedure.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.
- c. In addition to the calibration standards, analyze a quality control standard daily.
- d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- b. Set up and calibrate the instrument according to the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily.
- e. Analyze a duplicate sample daily.
- f. Use of the method of standard additions is recommended for all samples.

20. Analytical Balance

- a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.
- b. Weigh a standard weight each day the balance is used and maintain a log of values read.
- c. Check the balance zero before each use.
- d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

- a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.
- b. A copy of the approved reference procedures must be available in the laboratory.

22. Chemicals, Reagents, and Glassware

- a. Reagents must be prepared and used as detailed in the reference procedures.
- b. Date all chemicals received and all reagent solutions prepared.
- c. All chemicals should be reagent grade, when available.
- d. Maintain a record of all standardizations performed.
- e. We recommend that all glassware be Class A, when available.

23. Sample Preservation

- a. Samples should be preserved immediately after collection.
- b. Document the type of preservatives that are to be used and when samples are preserved.

24. Records

Analytical and quality control records must be available for inspection and include the following:

- a. Date samples are collected and date analyzed.
- b. Daily lab worksheets and workbooks.
- c. Values obtained on standards, blanks, duplicate samples, and standard curves.

- d. A record of all required quality controls.
- e. All worksheets must contain the signature or initials of the analyst(s) performing that function.
- f. All analytical records must be retained for at least three years.
- 25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed ± 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

a. Precisions Control Limits:

Using 30-40 sets of duplicate sample results or an annual data set, calculate precision control limits using the formulas given below:

Range (R) = 1st analysis - 2nd analysis

$$\overline{R} = \underline{\Sigma R}$$

$$\overline{n}$$

$$UWL_R = 2.51 \overline{R}$$

$$UCL_p = 3.27 \overline{R}$$

Where: \overline{R} = average range

UWL_p = Upper Warning Limit

UCL_p = Upper Control Limit

- 2.51 = Shewhart factor for 2s (duplicate)
- 3.27 = Shewhart factor for 3s (duplicate)
- NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1, 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.

- b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:
 - $\overline{P} = \frac{\text{observed}}{\text{known}} \times 100$ $\overline{\overline{P}} = \frac{\Sigma P}{n}$ $Sp = \sqrt{\frac{\Sigma P^2 (\Sigma P)^2}{n}}$ $UCL_p = \overline{P} + 3 Sp$ $UWLp = \overline{P} + 2 Sp$ $LCLp = \overline{P} 3 Sp$ $LWLp = \overline{P} 2 Sp$

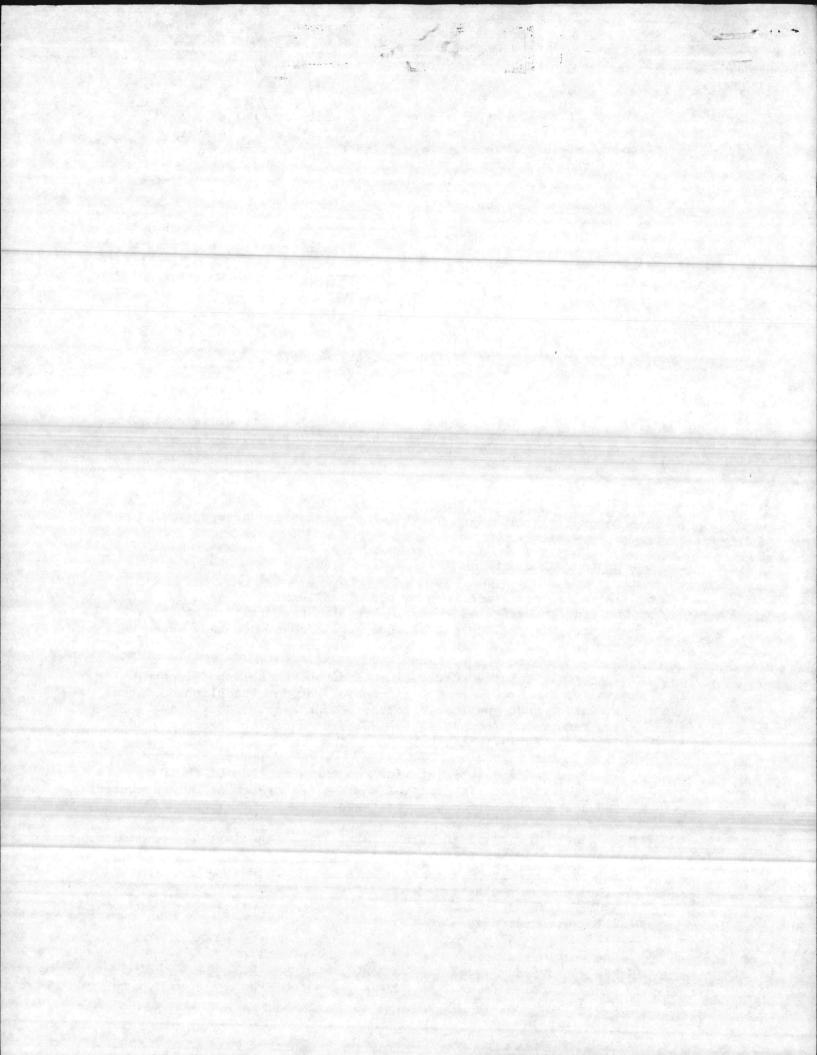
Where:

P	-	Percent recovery
P P	=	Average percent recovery
n	=	Number of analysis
Sp	=	Standard deviation of percent recovery
UCLp	=	Upper Control limit for percent recovery
UWLp	=	Upper warning limit for percent recovery
LCLp	=	Lower control limit for percent recovery
LWLp	=	Lower warning limit for percent recovery

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

- These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.
- (2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.
- (3) If seven successive points fall on the same side of the \overline{P} (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.
- e. For further information concerning statistical quality control limits, we recommend securing a copy of <u>EPA Handbook for Analytical Quality</u> <u>Control in Water and Wastewater Laboratories</u> EPA-600/4-79-019. This can be obtained by writing: Mr. Wade Knight Quality Assurance Officer U.S. EPA, Region 4 College Station Rd. Athens, GA 30613



F. D. Work Copy ...

Appears to contain section Info. Please refer to Prenous correspondence for Development Due Dates

27611

S. Thomas Rhodes, Secretary

Dear NPDES Permit Administrator:

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Quality Assurance Guidance and Steps Involved in Securing Certification RE:

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Enclosed is the quality assurance guidance that was promised in Mr. Helms' January 1985 letter that transmitted wastewater laboratory certification information. Part .0805(a)(6) of the certification regulation states that each laboratory shall develop and maintain a quality assurance document outlining the quality control practices of the laboratory. Laboratories are not expected to have a quality control document, but are expected to begin developing one.

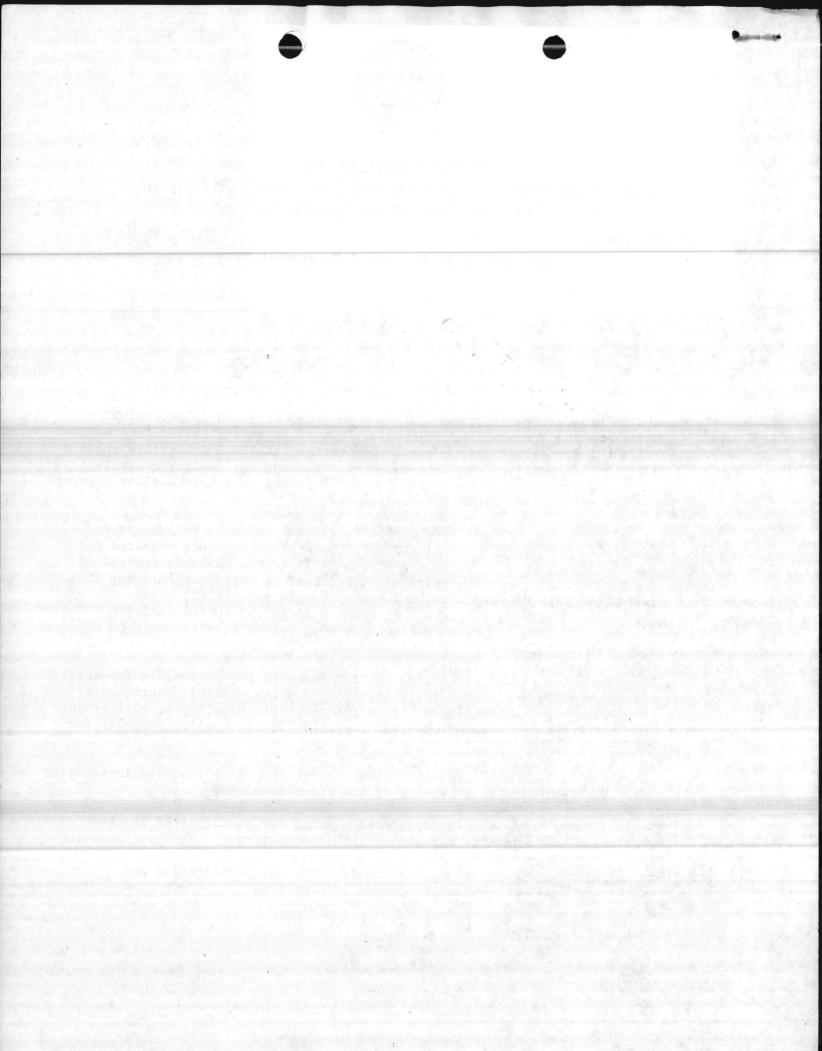
Parts I - III of the enclosed information is general guidance for developing a quality control plan and part IV is a listing of the minimum controls required for certification. Although it will be July or August 1985 before we begin processing certification applications, we encourage each laboratory to begin implementing the required quality controls so they will be better prepared for certification. We will consider substituting existing programs that are not identical to the enclosed required program. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

Questions have been raised concerning quality controls for parameters for which standards are not readily available (Ex. Coliform). Quality control requirements for these parameters have been modified. At a future public hearing, we will propose changes in the certification regulations that properly address quality controls for these parameters.

Some laboratories have requested information about the steps involved in securing certification. The following is a summary of the steps involved.

- The laboratory completes the application forms and submits them to the 1. Division of Environmental Management (DEM) Laboratory.
- 2. DEM Laboratory reviews the applications, notes any deficiencies and returns the application for any needed information.

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-4984





State of North Carolina Department of Natural Resources and Community Development 512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor

S. Thomas Rhodes, Secretary

Division of Environmental Management

Dear NPDES Permit Administrator:

RE: Quality Assurance Guidance and Steps Involved in Securing Certification

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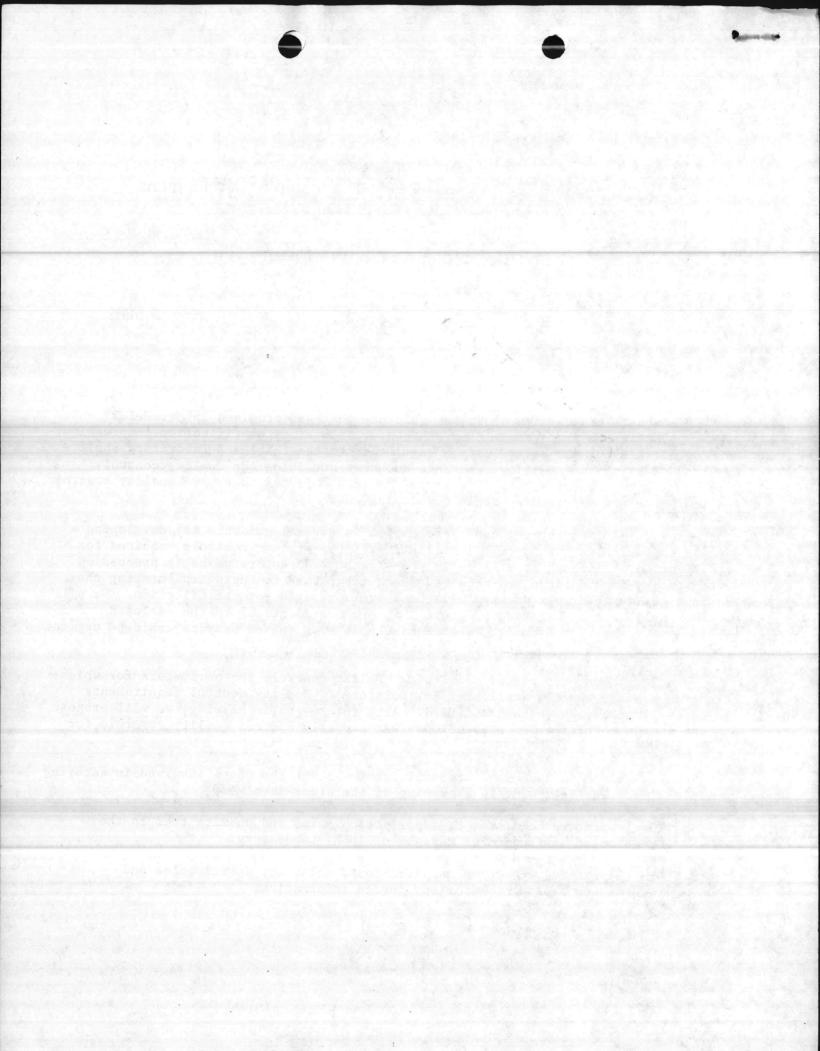
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P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-4984



NPDES Permit Administrator Page 2

- 3. DEM Laboratory mails performance evaluation samples to the laboratories seeking certification. This will begin in July or August 1985.
- 4. Your laboratory analyzes the samples and submits results to the DEM Laboratory.
- 5. DEM Laboratory reviews and evaluates these results and, if necessary, sends rerun samples. The DEM Laboratory will assist any laboratories having problems producing acceptable results. Assistance may be in the form of known samples, methodology, work forms, or quality controls.
- 6. The DEM Laboratory schedules and performs an onsite laboratory inspection. Time sensitive samples may be delivered during the inspection.
- 7. An inspection report listing any observed deficiencies will be prepared and mailed.
- 8. The laboratory seeking certification agrees to take any required corrective actions. Reasonable time will be given for taking corrective actions.
- 9. The laboratory requesting certification is billed the appropriate fees.
- 10. Upon receipt of payment, DEM issues certification for up to three years.

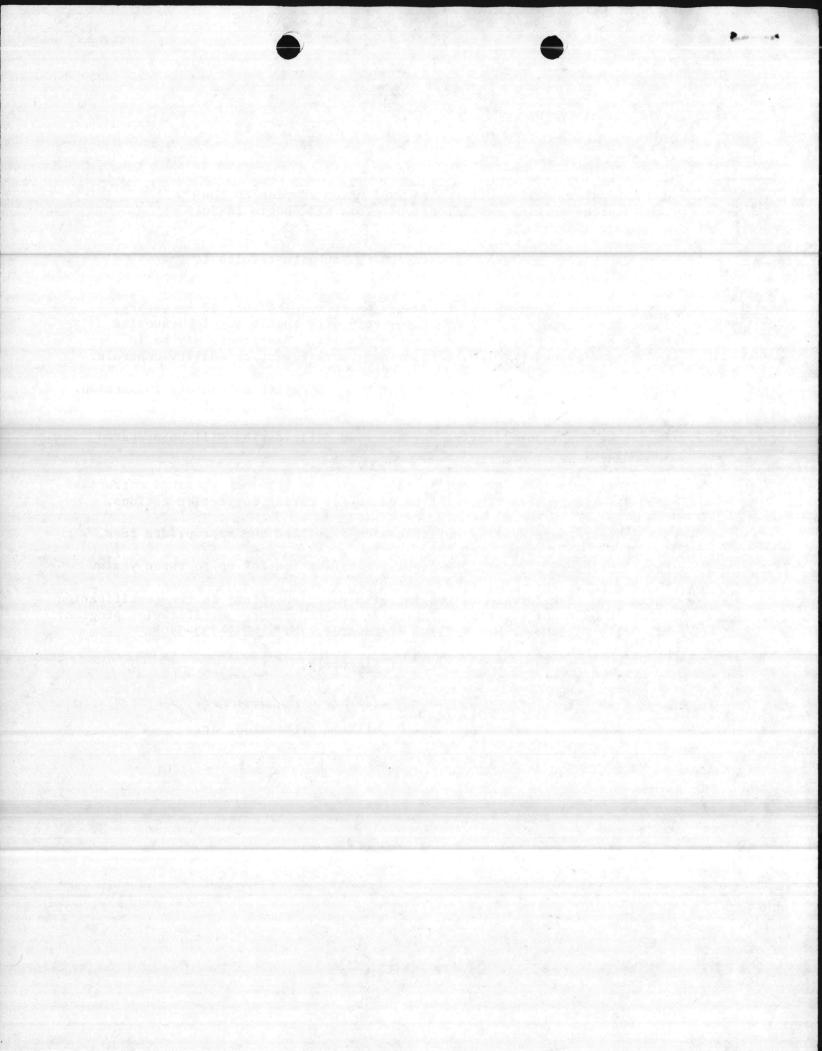
We continue to look forward to working with your laboratory in the certification program. If you have any questions concerning the laboratory certification program, contact Mr. Billy D. Byrd or Mr. William B. Edwards, Jr. at 919-733-3908.

Sincerely,

W. B. Edwards, gs.

William B. Edwards, Jr.

Enclosure



NRCD/DEM Wastewater Laboratory Certification Guidance for Preparing a Quality Assurance Document

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I. Introduction to moliperioo beas a biety of beas ad they been introduced

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

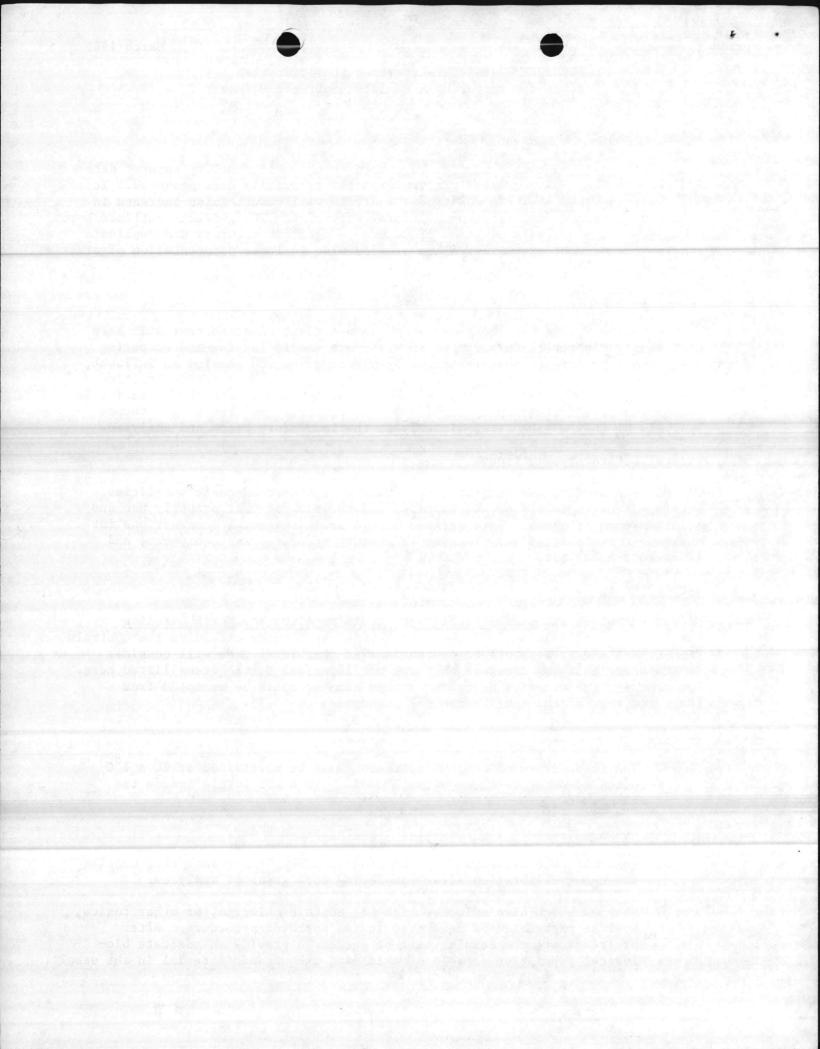
IV. Quality Controls syods add to be 0.01 saturing of ber.

Listed below are the <u>minimum quality controls required for North Carolina</u> <u>Wastewater Certification</u>. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

1. BOD

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- a. The temperature of the BOD incubator must be maintained at $20 \pm 1^{\circ}$ C using an accurate thermometer inserted in a BOD bottle inside the incubator.
- b. Check and record the incubator temperature each day.
- c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses.
- d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After **pretreatment**, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.



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- Perform a glucose-glutamic acid check each day seeded samples are analyzed. e.
- f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/l.
- Each day determine the BOD of the seed material the same as for any other g. unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.
- Analyze samples using a dilution series that will yield a dissolved h. oxygen usage of at least 2 mg/1 or a residual of at least 1 mg/1.
- Analyze a duplicate sample daily. appli al and any set at an al and i.
- Perform a blank dilution water control analysis along with each batch 1. of samples analyzed. as of you and the set you and the set of the
- COD Titration Procedure 2.
 - Standardize the COD titrant each day samples are analyzed. a.
 - Analyze a distilled water blank with each group of samples and make Ъ. proper corrections. Ceneral, Maharatany Prostices
 - Analyze a quality control standard along with each group of samples c. analyzed. Saving and the saligum but

Note: A 250 mg/1 COD standard may be made by dissolving .0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/l COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

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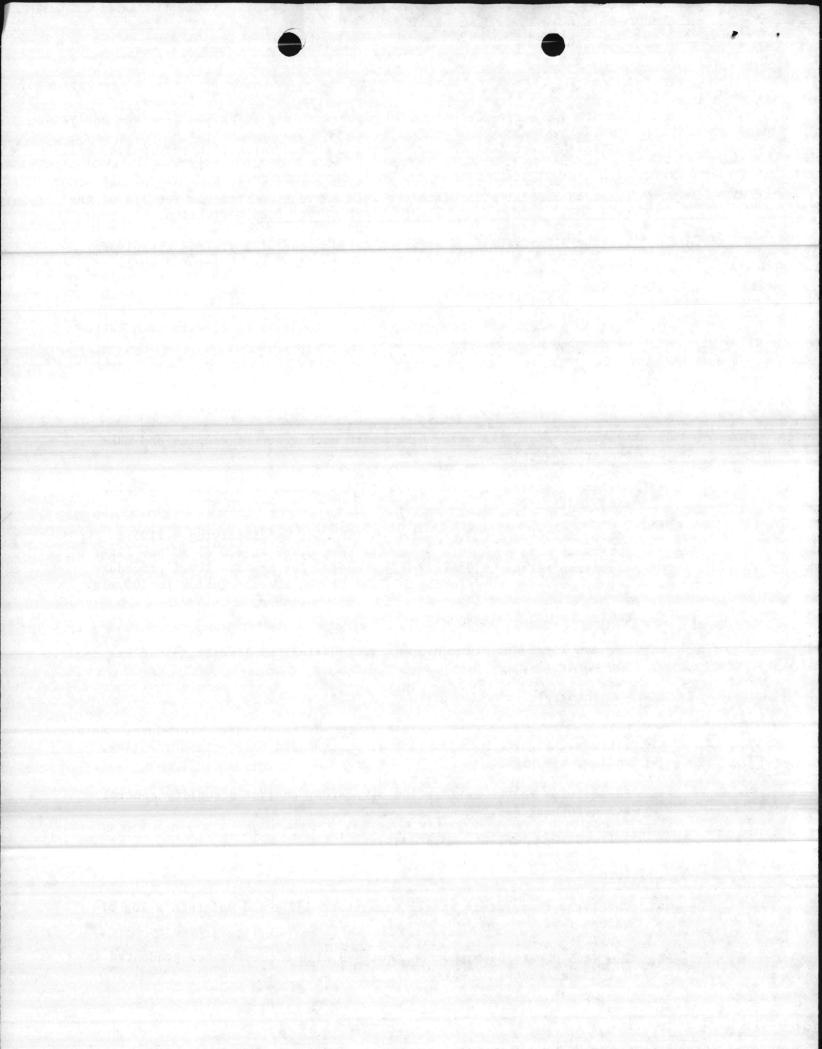
- d. Analyze a duplicate sample daily.
- Use the low level procedure for the analysis of samples with a COD of e. <50 mg/1. of factoredt ton ove fait scenning initialize substitutiedue

For enable lanceter of analyzing optical supples con 3. COD - Colormetric

Prepare a standard curve as set forth in the standard procedure. As a a. minimum. the curve must consist of a blank and three standards (low, medium, and high).

abrainers loring vities only is enor organizates.

- In addition to the calibration standards, analyze a quality control ь. standard each day.
- Analyze a duplicate sample daily. c.
- c. Calibrate the classived orygen, mater each day borors Coliform to quote sufficientou after completing such group of
 - Check the temperature of all incubators daily and maintain a log of a. values read, worroch sits of badinoash as letter and data
 - a of osbess ad term astimut The 44.5°C waterbath must be equipped with a thermometer graduated in Ъ. 0.1°C increments.



- c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.
- d. Log the maximum temperature and pressure of the autoclave once during each use.
- e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.
- f. Analyze one duplicate sample each day.

5. Chloride

a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.

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- b. Analyze a distilled water blank each day and make proper corrections.
- c. Analyze one duplicate sample each day.

6. Hardness

- a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.
- b. Analyze a distilled water blank each day samples are analyzed.

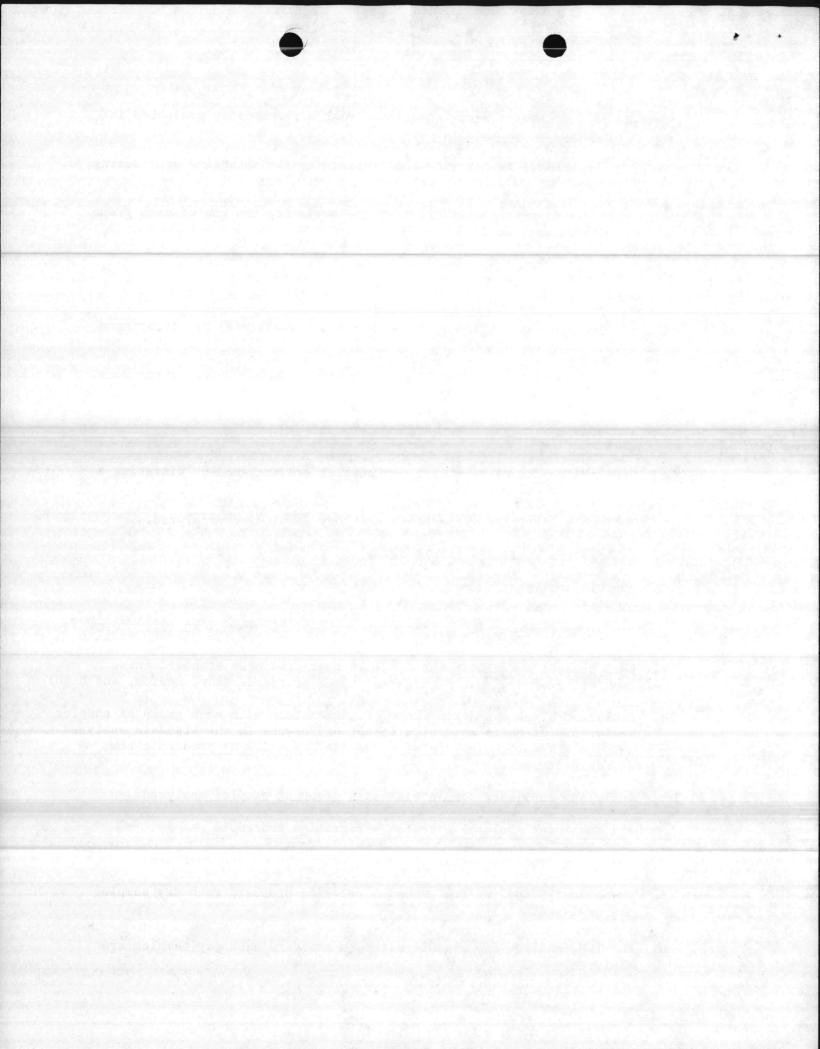
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- c. Analyze one duplicate sample daily.
- 7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

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- a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).
- b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.
- c. Analyze a duplicate sample with each group of samples analyzed.
- 8. Ammonia and Total Kjeldahl Nitrogen Titration Procedure
 - a. Analyze a distilled water blank each day samples are analyzed.
 - b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.
 - c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.



Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing l ml concentrated H₂SO₄ and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated H₂SO₄ to one liter with distilled water will yield a solution containing² 1 mg/l of nitrogen.

- d. Analyze a duplicate sample daily. redail of their and sha
- 9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

- a. Calibrate the meter according to the manufacturer's instructions.
- b. Check the meter calibration by analyzing a medium level quality control standard each day.
- c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

Station from

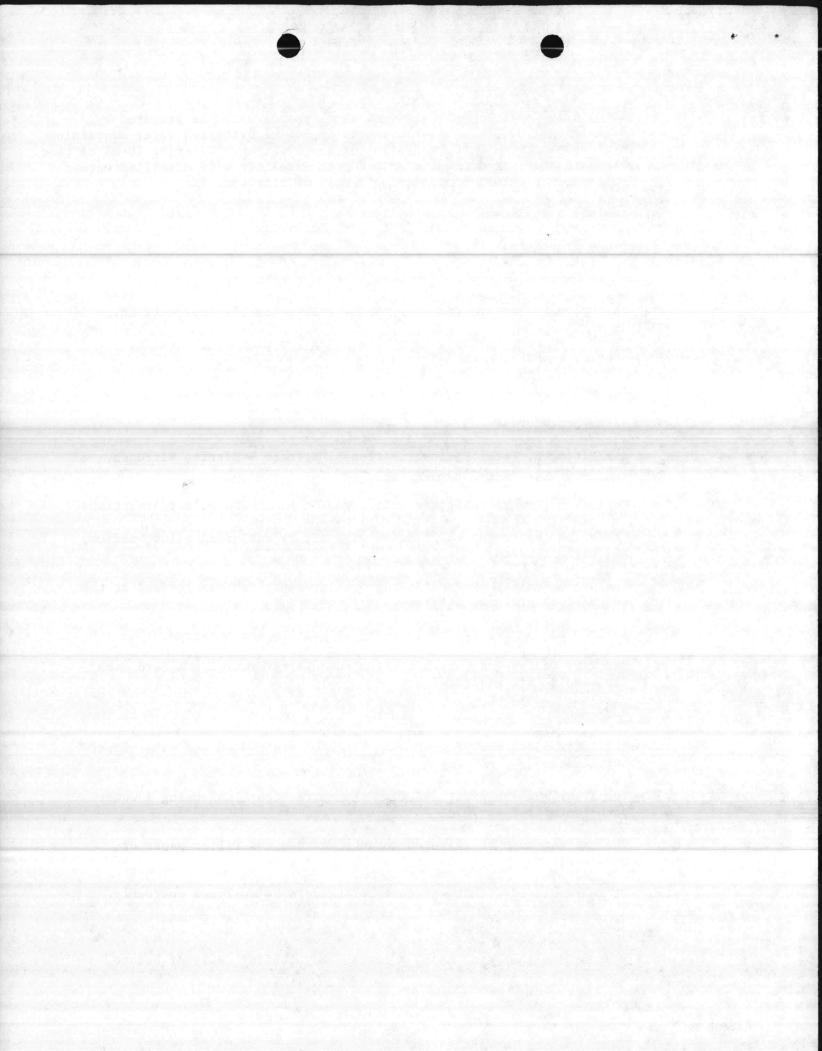
Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

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- a. Calibrate the instrument according to the manufacturer's instructions.
- b. Check the instrument calibration each day by analyzing a low, medium, and high standard.
- c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.
- d. Analyze one duplicate sample each day samples are analyzed.
- e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

- a. Perform a blank analysis on each batch of freon used and make proper corrections.
- b. The freen must be distilled from the extraction flask using a water bath controlled at 70°C.
- c. The extract must be filtered through Whatman #40 filter paper or equivalent.
- d. It is recommended that a reference standard be analyzed quarterly.
- 12. pH
 - a. Standardize the meter using a low and high buffer daily or before each use.

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- b. It is recommended that a reference standard be analyzed quarterly.
- c. Analyze a duplicate sample daily.
- 13. Total Residue and Total Suspended Residue
 - a. Check and record drying oven temperature each day used.
 - b. Analyze one duplicate sample each day samples are analyzed.
 - c. It is recommended that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.
 - d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

- a. Standards as described in the approved procedure must be secured and used.
- b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.
- c. Analyze one duplicate sample each day samples are analyzed.
- d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

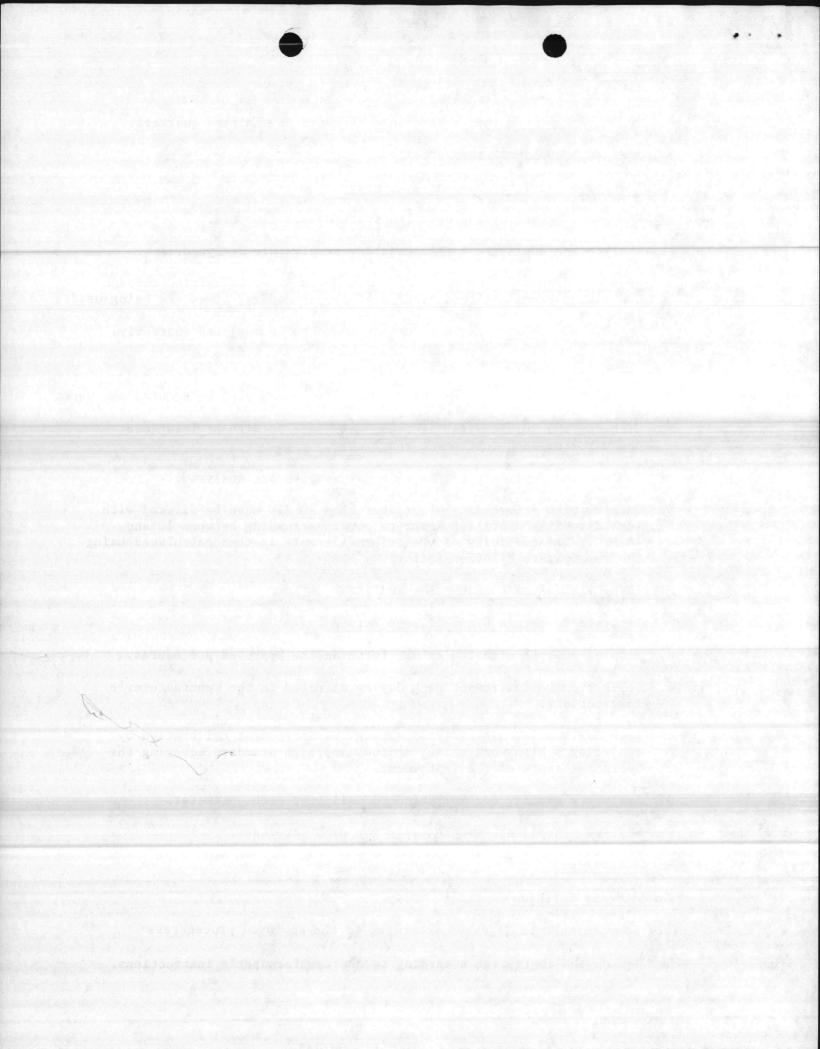
Metals Group I, Metals Group II, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- b. Calibrate the instrument each day as directed in the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily for each parameter.
- e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

- a. Samples must be digested according to the approved procedures.
- b. Set up the instrument according to the manufacturer's instructions.



c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.

-6-

- d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.
- e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

- a. Samples must be digested according to the approved procedures.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.
- c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.

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d. Analyze one duplicate sample daily.

18. Mercury

- a. Set up the instrument according to the manufacturer's instructions and the approved procedure.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.
- c. In addition to the calibration standards, analyze a quality control standard daily.

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d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

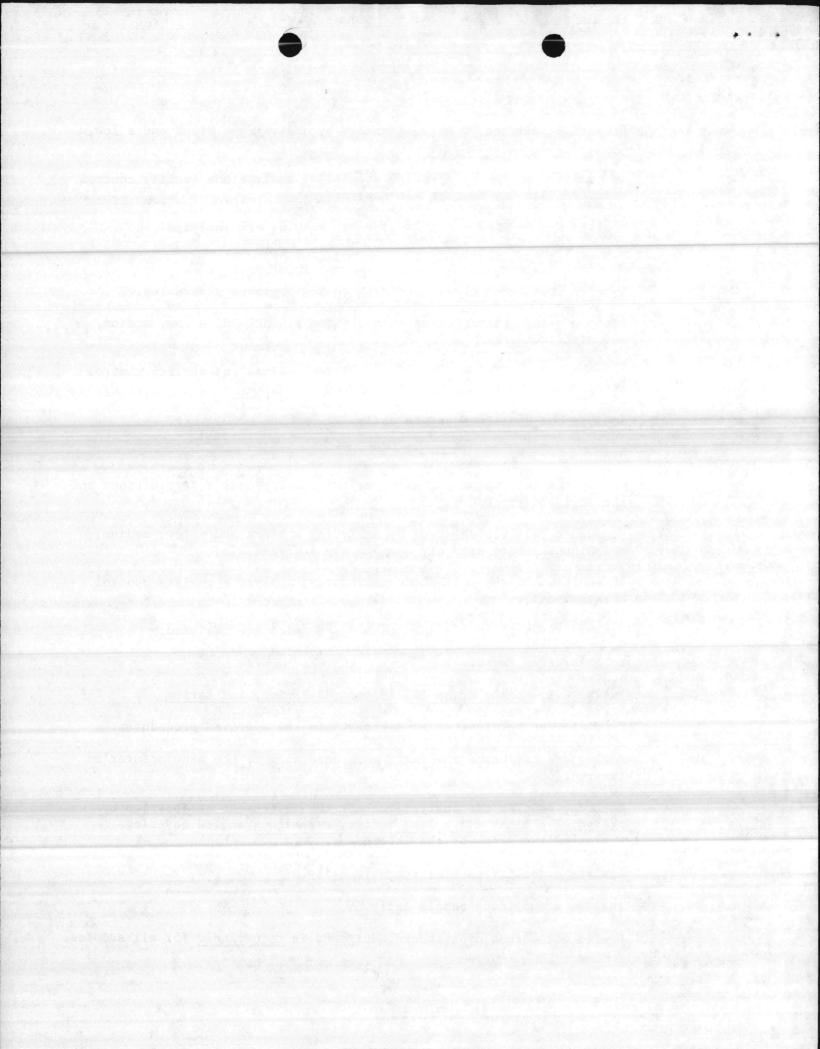
Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- b. Set up and calibrate the instrument according to the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily.

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- e. Analyze a duplicate sample daily.
- f. Use of the method of standard additions is recommended for all samples.

16.4 199



20. Analytical Balance

- a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.
- b. Weigh a standard weight each day the balance is used and maintain a log of values read.
- c. Check the balance zero before each use.
- d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

- a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.
- b. A copy of the approved reference procedures must be available in the laboratory.

22. Chemicals, Reagents, and Glassware

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- a. Reagents must be prepared and used as detailed in the reference procedures.
- b. Date all chemicals received and all reagent solutions prepared.
- c. All chemicals should be reagent grade, when available.
- d. Maintain a record of all standardizations performed.
- e. We recommend that all glassware be Class A, when available.

23. Sample Preservation

- a. Samples should be preserved immediately after collection.
- b. Document the type of preservatives that are to be used and when samples are preserved.

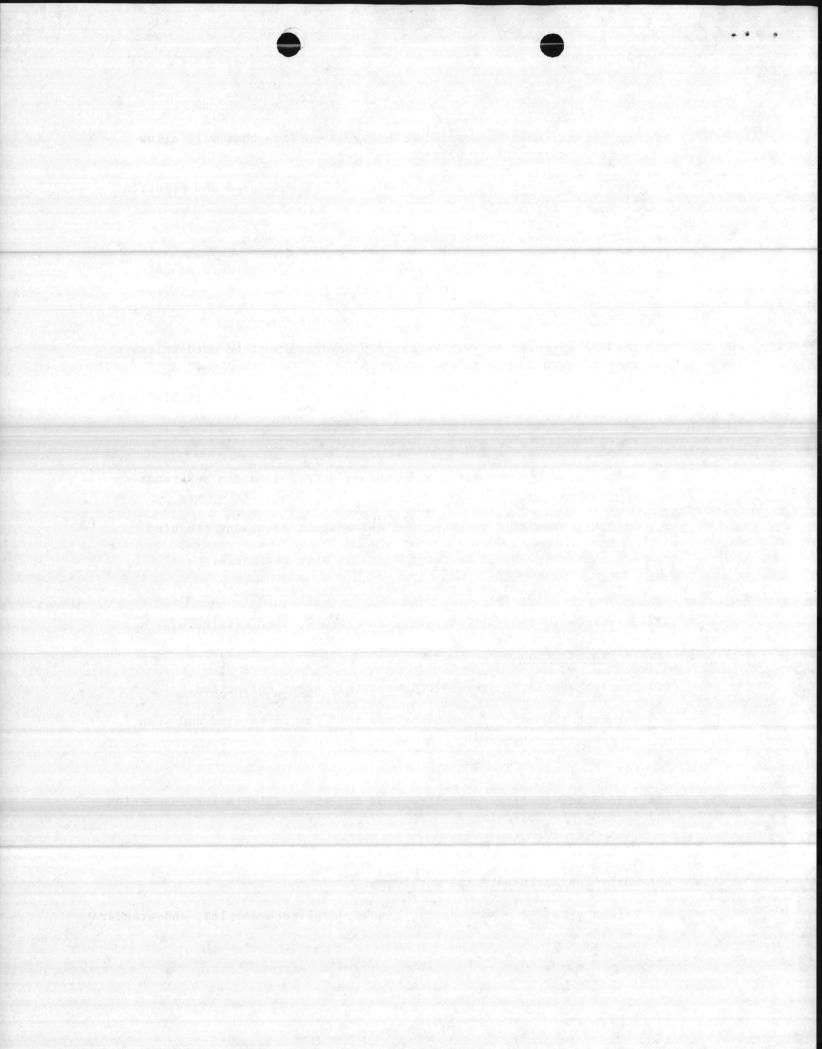
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- d. A record of all required quality controls.
- e. All worksheets must contain the signature or initials of the analyst(s) performing that function.
- f. All analytical records must be retained for at least three years.

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25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed \pm 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

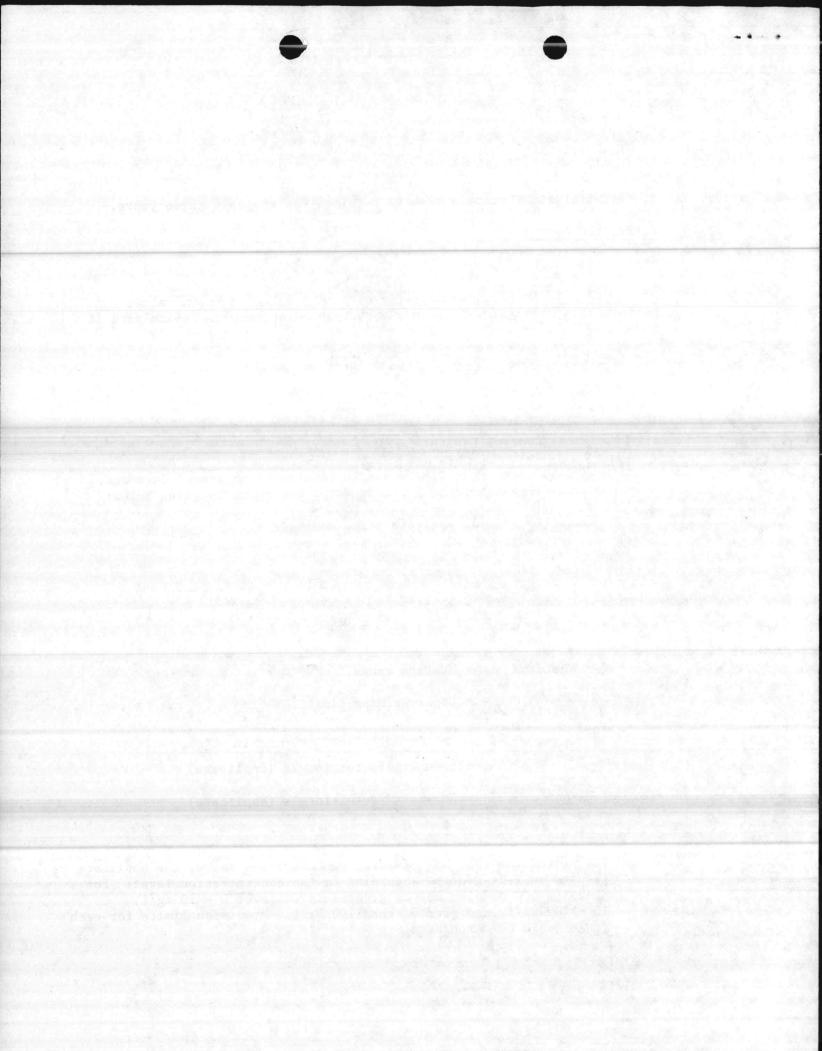
It is <u>recommended</u> that each laboratory calculate statistical control limits, but it is <u>not required</u> at this time.

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		= 3.27 R
Where:	R	= average range
righting a	UWLR	Upper Warning Limit
hear ea os	UCLR	= Upper Control Limit
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NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1, 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.



b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

Sp-

- P = <u>observed</u> x 100 known
- $P = \Sigma P$

	UCL	-	P + 3 Sp
$\sqrt{\frac{\Sigma P^2 - (\Sigma P)^2}{n}}$	UWLP	-	P + 2 Sp
$\sqrt{\frac{n}{n-1}}$	LCLp	.=	P - 3 Sp
	LWLp	-	₽ - 2 Sp

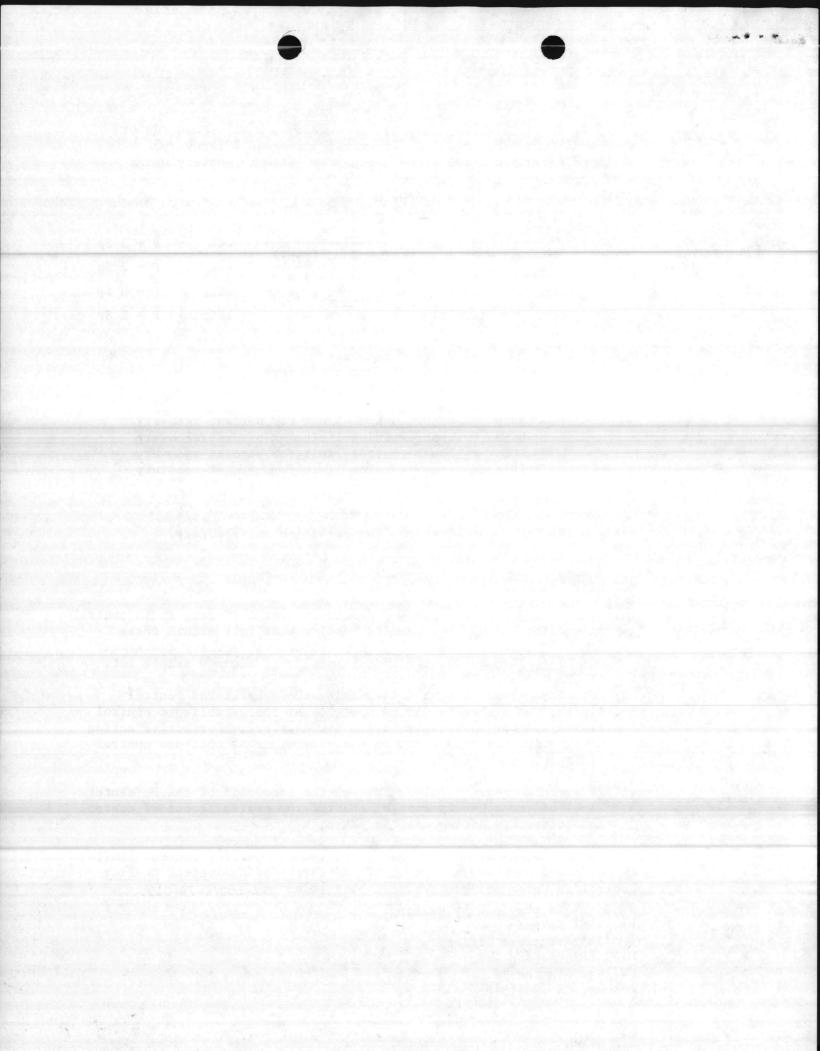
Where:

PP	-	Percent recovery
P	-	Average percent recovery
n	-	Number of analysis
Sp	-	Standard deviation of percent retovery
UCLp	=	Upper Control limit for percent recovery
UWLp	-	Upper warning limit for percent recovery
LCLp	-	Lower control limit for percent recovery
LWLp	=	Lower warning limit for percent recovery
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c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

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- (2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.
- (3) If seven successive points fall on the same side of the P (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.
- e. For further information concerning statistical quality control limits, we recommend securing a copy of <u>EPA Handbook for Analytical Quality</u> <u>Control in Water and Wastewater Laboratories</u> EPA-600/4-79-019. This can be obtained by writing: Mr. Wade Knight Quality Assurance Officer U.S. EPA, Region 4 College Station Rd. Athens, GA 30613





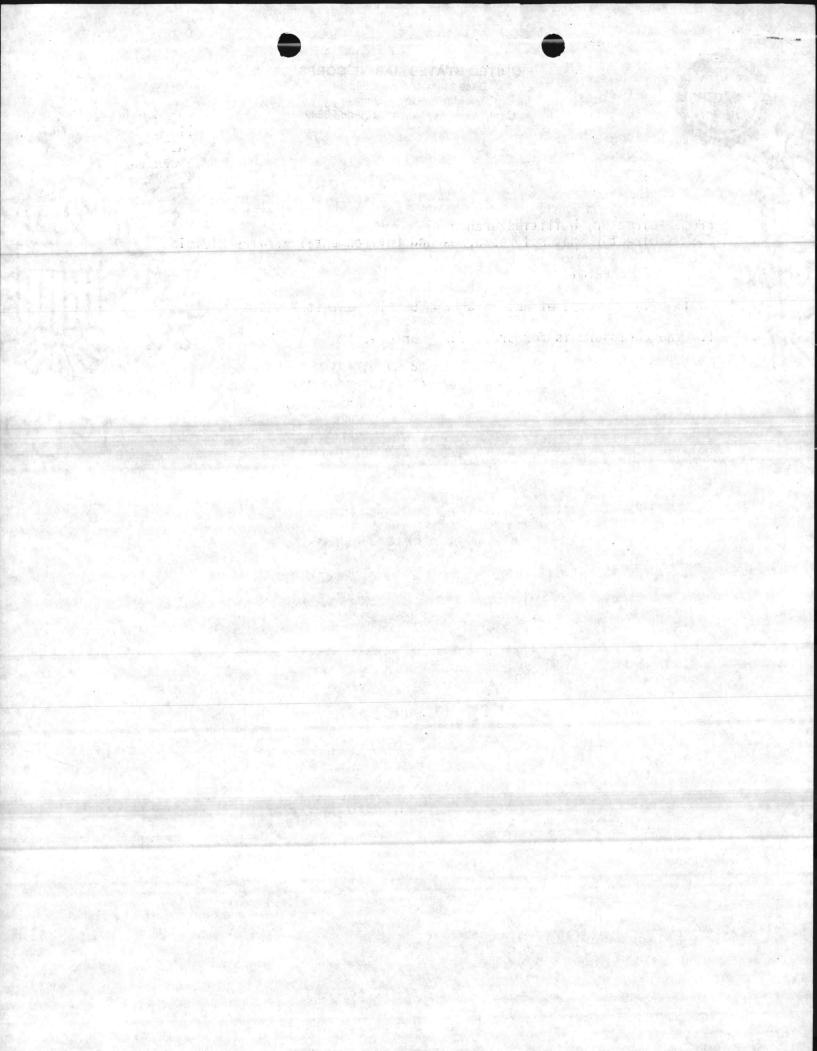
UNITED STATES MARINE CORPS Base Maintenance Division Marine Corps Base Camp Lejeune, North Carolina 28542

in reply refer to 11330 MAIN 4 Jan 85

From: Director, Utilities Branch To: Director, Natural Resources and Environmental Affairs Division Subj: NPDES Permit Encl: (1) NC Dept of NatRes & Comm Development 1tr undtd

1. The enclosure is forwarded for your use.

M.S. Johnson, JR.



NREA



North Carolina Department of Natural Resources & Community Development

James B. Hunt, Jr., Governor

James A. Summers, Secretary

DIVISION OF ENVIRONMENTAL MANAGEMENT

> Robert F. Helms Director

Telephone 919 733-7015

Dear NPDES Permit Administrator:

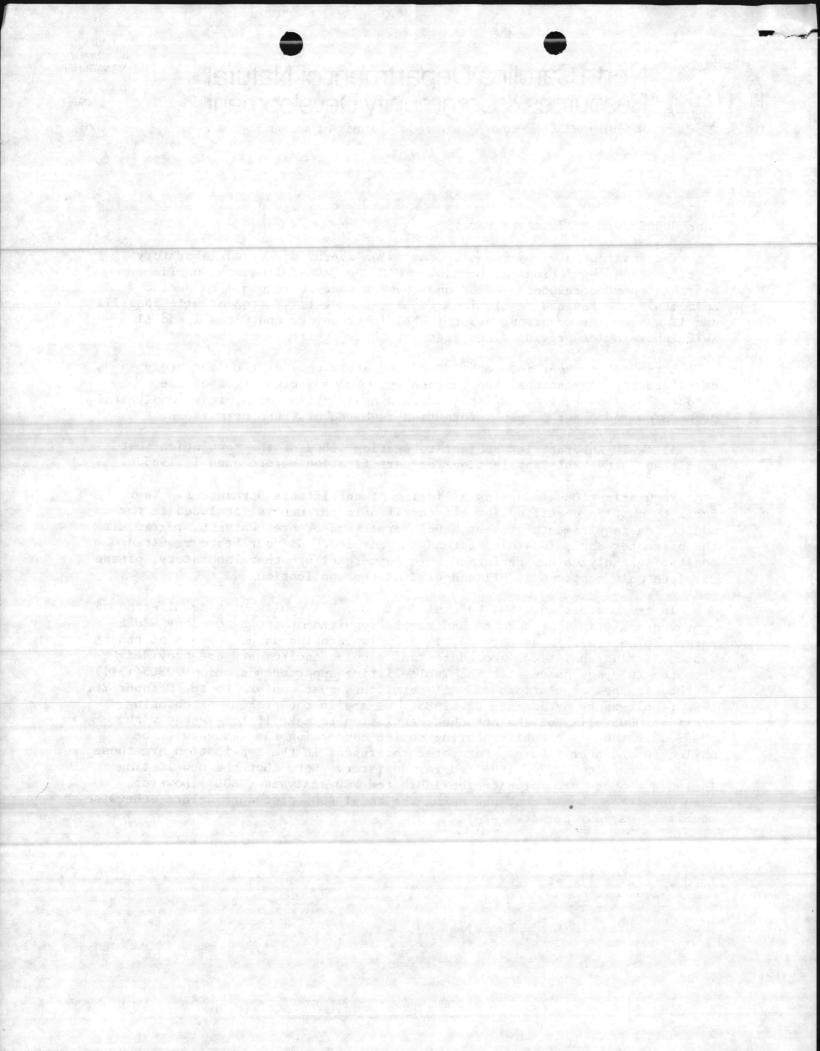
The Environmental Management Commission approved revised laboratory certification regulations as found in NCAC 2H .0800 following a public hearing, comment response period and changes made in response to public comments. The revised regulations require the certification of all Class III and IV wastewater treatment plant (WWTP) laboratories and Class I and II WWTP laboratories providing analyses for their pretreatment programs.

Attached for your information and use are a copy of the laboratory certification regulations, application form and selected pages of the October 26, 1984 Federal Register containing a listing of approved analytical procedures and other sample information required of NPDES permittees.

All WWTP Laboratories subject to section .0802 of the regulation must submit an acceptable application for certification before June 1, 1985.

Regulation .0804 contains a listing of certifiable parameters. Your facility must be certified for all certifiable parameters, included in the monitoring requirements of your NPDES Permit and/or pretreatment program, if the parameter analysis are performed in your lab. If your State required monitoring analyses are performed by a commercial or other laboratory, please complete only sections I, VII and VIII of the application.

In the October 26, 1984 Federal Register, EPA published a new listing of approved analytical procedures and sample requirements to be used by NPDES permit holders with a January 24, 1985 implementation date. Copies of the Tables listing analytical procedures and sample requirements are provided from the 203 page document. The Table listing <u>supersedes</u> section .0805(a)(1) of the attached regulations and all permittees must conform to the October 26, 1984 requirements by January 24, 1985. We are in the process of changing section .0805(a)(1) of the attached regulation to make it consistent with EPA requirements. A public hearing notice concerning the change will be mailed in the near future. Parameter references in the application are those cited in the October 26, 1984 Federal Register. Note that the new listing includes a procedure for nitrogen inhibited BOD analysis (CBOD₅), however, each permittee should continue using the total BOD₅ procedure unless otherwise specified on your permit.



NPDES Permit Administrator Page 2

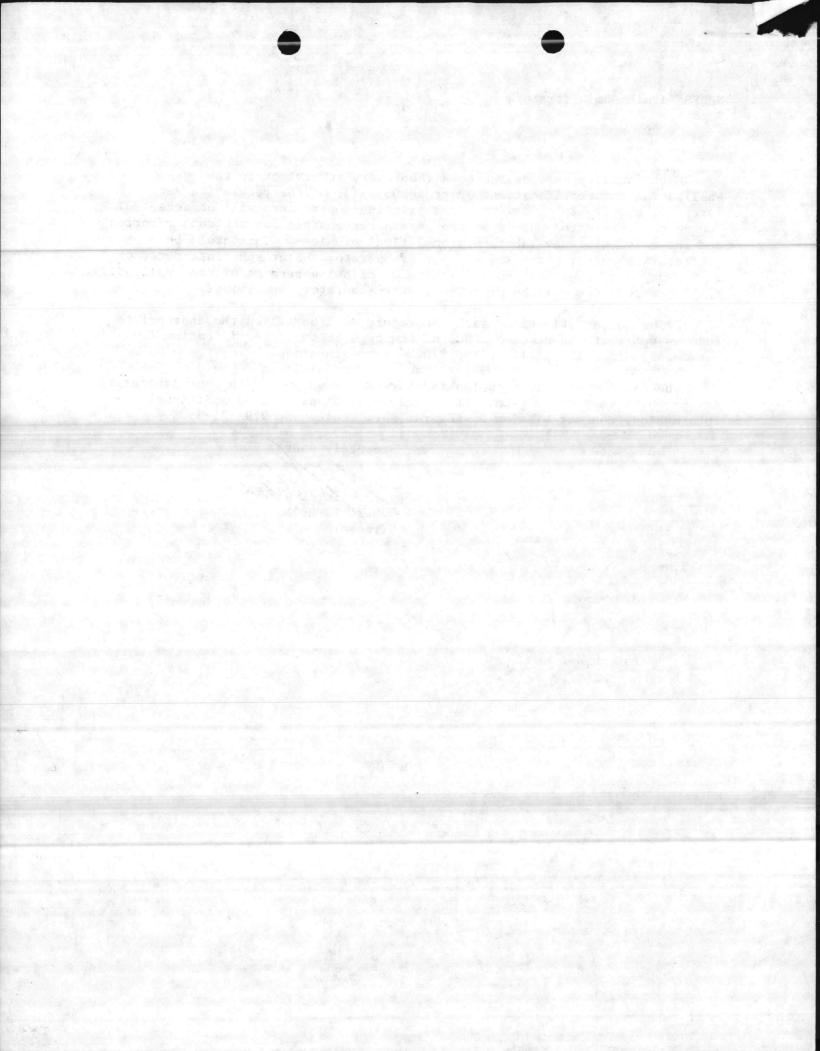
Each facility must designate a laboratory supervisor in the application for certification. Part .0805(a)(3) of the regulation sets forth the requirements, however, all existing supervisors will be accepted. The person designated should be the person responsible for the data reported and the solving of any laboratory analytical problems. This could be a laboratory chemist, plant manager, chief operator, plant superintendent, or City manager if their duties so dictate. Following receipt of the application, all correspondence will be directed to the laboratory supervisor.

Prior to certification, each laboratory will be billed the appropriate fees as set forth in section .0806 of the regulations. Certification fees for metals Group I and II will be \$20.00 for each group.

The Laboratory Branch is looking forward to working with your laboratory in the certification program. If you have questions or need additional information, contact William B. Edwards, Jr., telephone 919-733-3908.

Sincerely your, Robert F. Helms

Robert F. Helf Director





North Carolina Department of Natural Resources & Community Development

James B. Hunt, Jr., Governor

James A. Summers, Secretary

DIVISION OF ENVIRONMENTAL MANAGEMENT

> Robert F. Helms Director

Telephone 919 733-7015

Dear NPDES Permit Administrator:

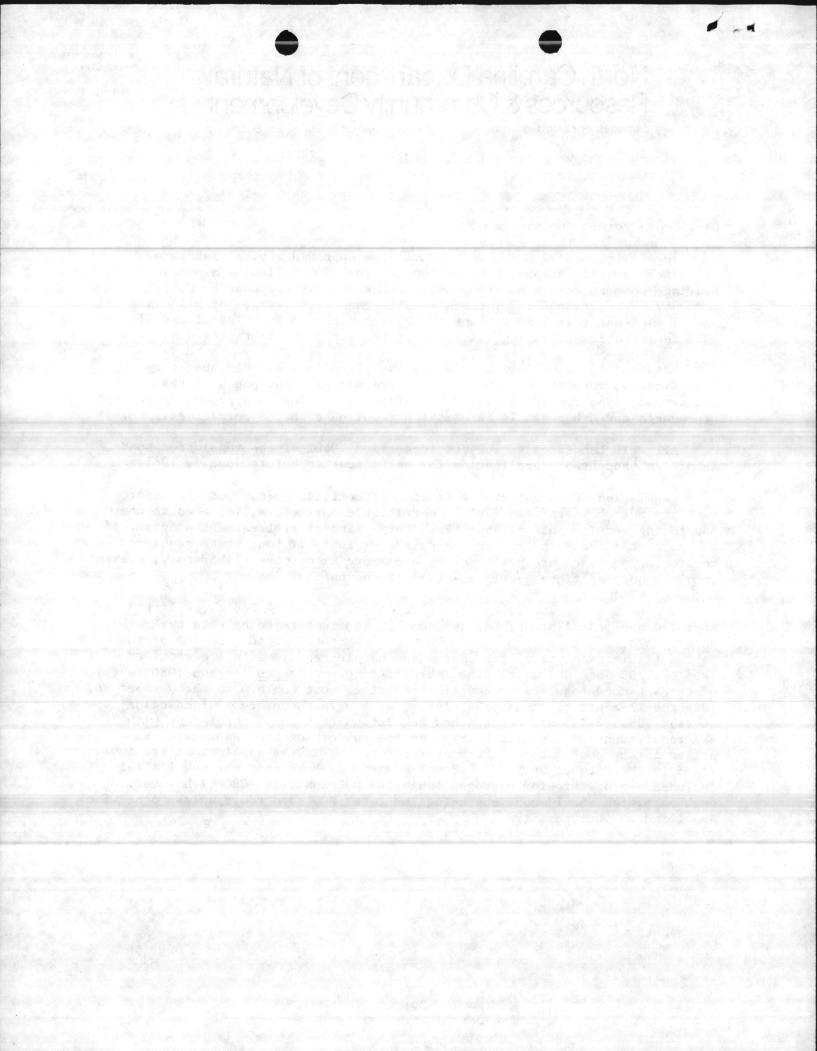
The Environmental Management Commission approved revised laboratory certification regulations as found in NCAC 2H .0800 following a public hearing, comment response period and changes made in response to public comments. The revised regulations require the certification of all Class III and IV wastewater treatment plant (WWTP) laboratories and Class I and II WWTP laboratories providing analyses for their pretreatment programs.

Attached for your information and use are a copy of the laboratory certification regulations, application form and selected pages of the October 26, 1984 Federal Register containing a listing of approved analytical procedures and other sample information required of NPDES permittees.

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NPDES Permit Administrator Page 2

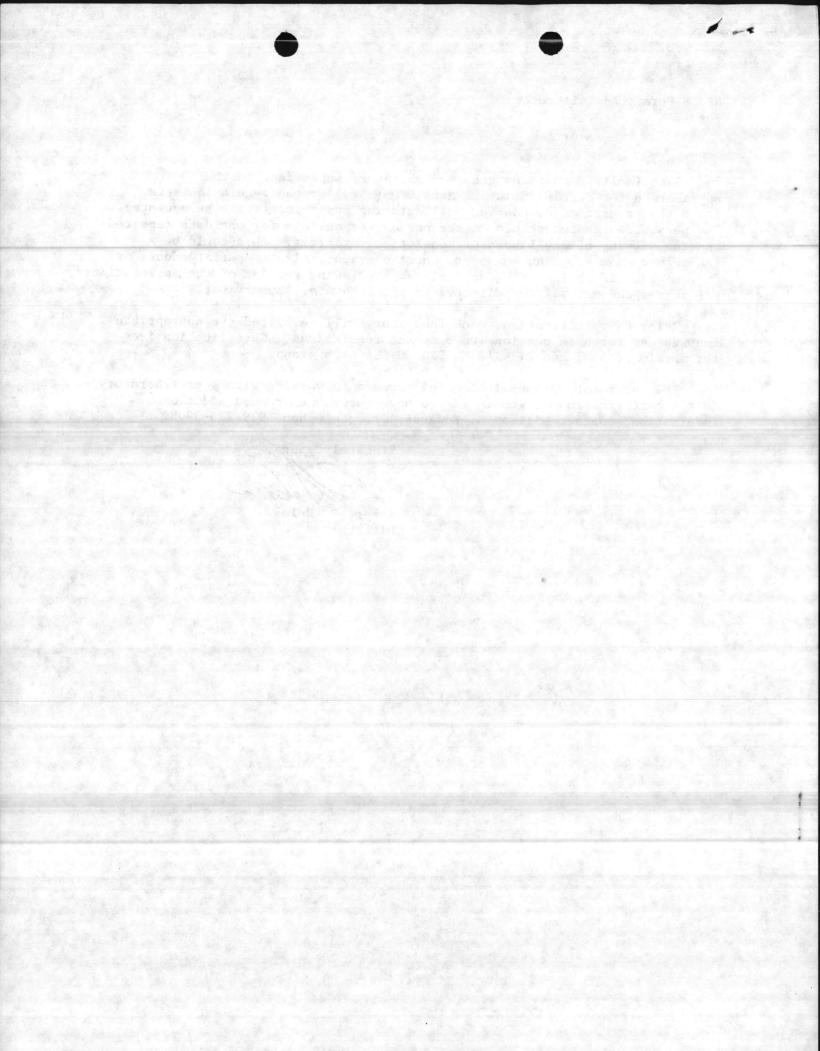
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NORTH CARÓLINA ADMINISTRATIVE CODE

TITLE 15

DEPARTMENT OF NATURAL RESOURCES

AND

COMMUNITY DEVELOPMENT

CHAPTER 2

ENVIRONMENTAL MANAGEMENT DIVISION

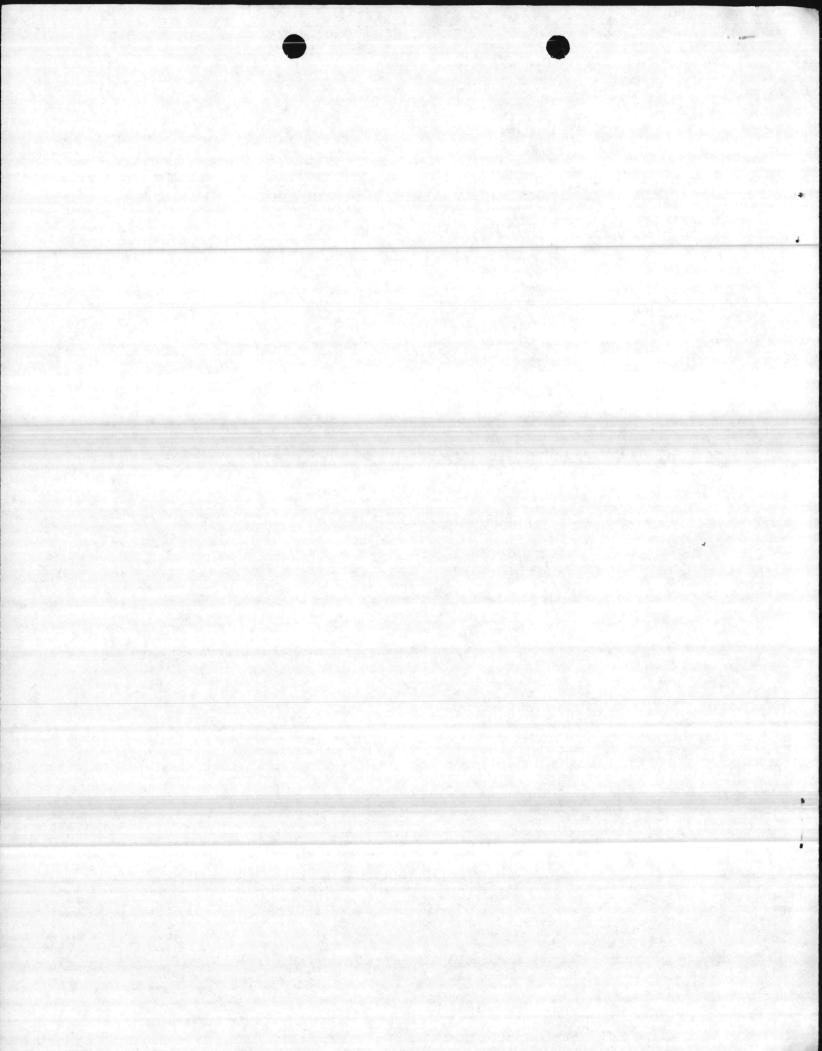
SUBCHAPTER 2H

PROCEDURES FOR PERMITS, APPROVALS SECTION .0800 LABORATORY CERTIFICATION



This copy has been officially filed with the Attorney General. Current through December 1, 1984.

ENVIRONMENTAL MANAGEMENT COMMISSION RALEIGH, NORTH CAROLINA



NRACD - ENVIRONMENTAL MANAGEMENT

T15: 02H .0800

SECTION .0800 - LABORATORY CERTIFICATION

64.7

64.9

64.25

. 0801 PURPOSE

These Regulations set forth the requirements for state 64.11 certification of commercial, municipal, and industrial 64.12 laboratories to perform water analyses, required by the Water and Air Quality Reporting Act, G.S. 143-215.63 et seq: Environmental 64.13 64.14 64.15 Management Commission Regulations for Surface Water Monitoring, 64.16 Reporting, found in Subchapter 2B of this Chapter, Section .0500 and Environmental Management Commission Regulations for Local 64.17 Pretreatment Programs, found in 15 NCAC 2H .0900.

History Note:	Statutory Authority G.S. 143-215.3(a) (1);	64.20
	143-215.3(a)(10); Eff. February 1, 1976;	64.22
	Amendel Eff. December 1, 1984; November 1, 1978.	64.23

.090? SCOPE These Regulations apply to commercial laboratories and Class 64.27 III and IV municipal or industrial wastewater treatment plant 64.28 laboratories which perform water analyses for persons subject to 64.29 G.S. 143-215.1, 143-215.63, et seq., or the Environmental 64.30 Management Commission Regulations for Surface Water Monitoring, 64.32 Reporting found in Subchapter 2B of this Chapter, Section .0500. 64.33 These Regulations also apply to all wastewater treatment plant 64.34 laboratories for municipalities having Local Pretreatment Programs as found in 15 NCAC 2H .0900. Municipal and industrial 64.35 laboratories that perform analyses for two or less of the 64.36 parameters listed in Paragraph .0804 (a) of these Regulations are 64.37 exempt from the requirements of these Regulations. 64.38

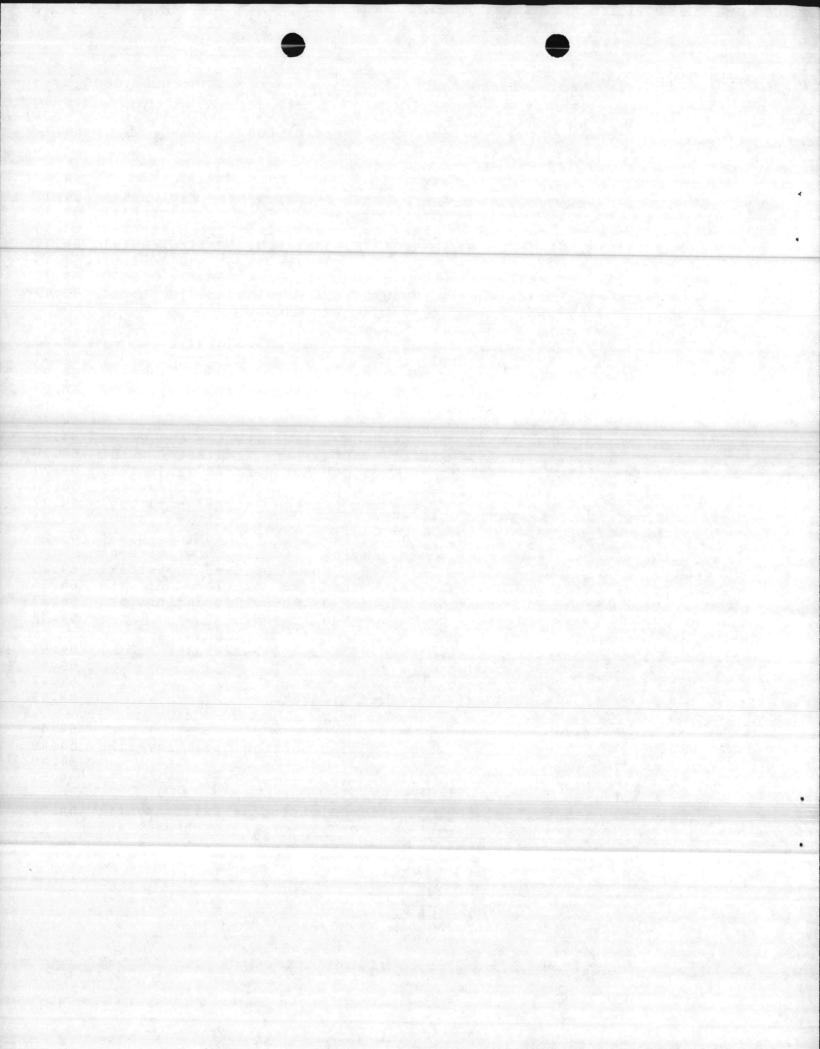
History Note:	Statutory Authority G.S. 143-215.3(a) (1);	64.41
history no ce.	143-215. 3 (a) (10) ;	64.42
	Eff. February 1, 1976;	64.43
	Amended Eff. December 1, 1984.	64.44

64.46 . 1907 DEFINITIONS The following terms as used in this Section shall have the 64.48 64.49 essigned meaning: 64.52

- Laboratory means any laboratory which is (1) Commercial seeking to analyze water samples for others.
- (?) State means the North Carolina Division of Environmental 64.54 Management of the Department of Natural Resources and 64.55 Community Development, or its successor.

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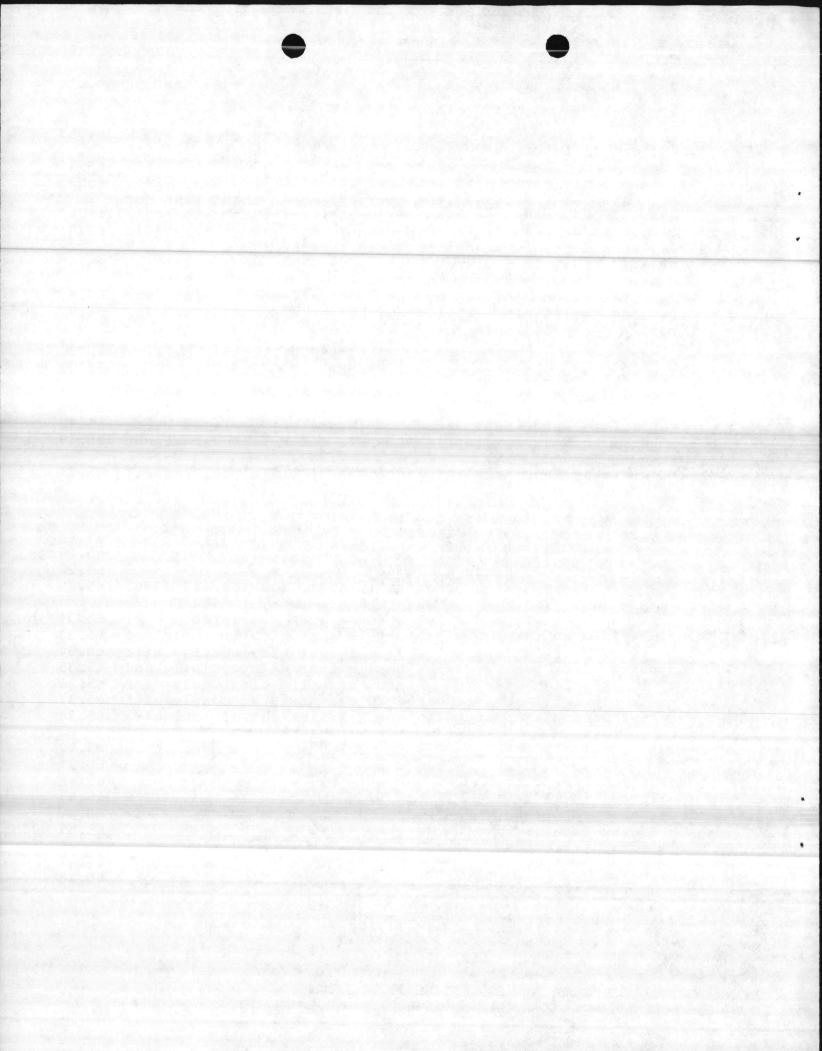


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(3)	State Laboratory means the laboratory branch of the North	64.57
	Carolina Division of Environmental Management, or its	1
	successor.	65.1
(4)	Unacceptable results on performance evaluation samples or	65.3
	split samples are those that vary by more than plus or	
	minus 25 percent of the value determined by the State	65.4
	Laboratory or the State Laboratory may adopt specific	65.5
	variance limits for a particular parameter.	
(5)	Certification is a declaration by the state that the	65.7
	personnel, equipment, records, guality control procedures,	
	and methodology cited by the applicant are accurate and	65.8
	that the applicant's proficiency has been considered and	65.9
	found to be acceptable.	
(6)	Decertification is loss of certification.	65.11
(-)	Recertification is reaffirmation of certification.	65.12
(8)	Sunicipal Laboratory means a laboratory operated by a	65.15
7.07	municipality or other local government to analyze samples	Martin .
	from its wastewater treatment plant (s).	65.16
(?)	Industrial Laboratory means a laboratory operated by an	65.18
7.1	industry to analyze samples from its wastewater treatment	65.19
	plant (s).	05.15
(10)	Pretreatment Program means a program of waste pretreatment	65.21
(10)	requirements set up in accordance with 15 NCAC 2H .0900	03.21
	requirements set up in accordance with 15 wike 24 .0900	65.22
	and approved by the Division of Environmental Management.	03.44
Hist	ory Note: Statutory Authority G.S. 143-215.3(a) (1);	65.26
	143-215.3 (a) (10);	65.27
	Eff. February 1, 1976;	65.28
	Amended Eff. December 1, 1984; November 1, 1978.	65.29
.0804	PAPAMETERS FOR WHICH CERTIFICATION NAY BE REQUESTED	65.31
	nercial laboratories need to obtain certification only for	65.33
	ters which will be reported by the client to comply with	65.34
	monitoring and pretreatment regulations. Municipal and	65.35
	rial Laboratories need to obtain certification only for	65.36
	ters which will be reported to the state to comply with	65.37
	oring and pretreatment regulations. A listing of selected	65.38
	eters follows:	
(1)	BOD	65.40
(2)	COD	65.41
	Chloride	65.42
(7)		65.43
(")	Coliform, fecal MF	
(5)	Coliform, total MF	65.44
151	Coliform, fecal tube	65.45
(~)	Coliform, total tube	65.46
(8)	Cyanide	65.47
(\circ)	Fluoride	65.48

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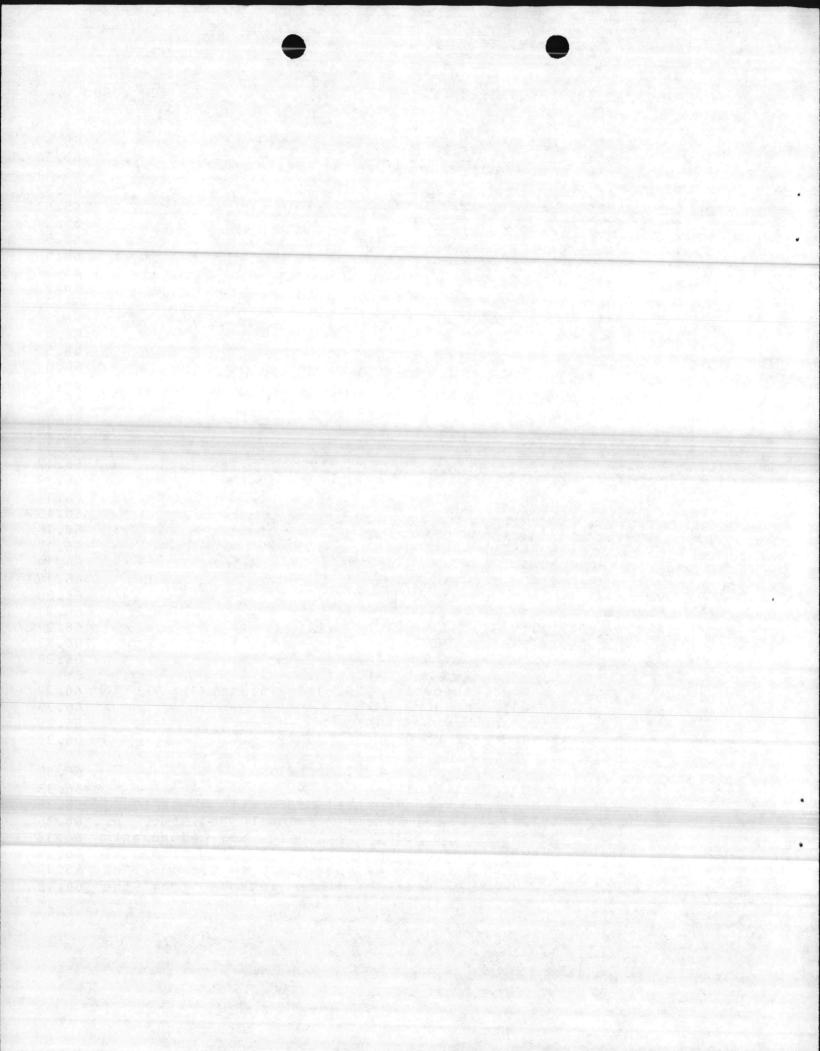


NPSCD - ENVIPONMENTAL MANAGEMENT

T15: 02H .0800

(10) Grease and Oil	65.49
(11) Hardness, total	65.50
(12) MBAS	65.51
(13) Metals, Group I	65.52
(a) aluminum	65.53
(b) beryllium	65.54
(c) cadmium	65.55
(d) chromium, total	65.56
(e) cobalt	65.57
(f) copper	66.1
(g) iron	66.2
(h) lead	66.3
(i) maganese	66.4
(j) nickel	66.5
(k) zinc	66.6
(14) Metals, Group II	66.7
(a) antimony	66.8
(b) silver	66.9
(c) thallium	66.10
(1 ⁵) Arsenic	66.11
(16) Barium	66.12
(17) Mercury	66.13
(1°) Selenium	66.14
(19) Ammonia nitrogen	66.15
(70) Total Kjeldahl nitrogen (TKN)	66.16
	66.17
(22) Total phosphorus	66.18
(27) Orthophosphate	66.19
	66.20
(25) Phenols	66.21
(26) Residue, total	66.22
(27) Residue, total suspended	66.23
(28) Turbidity	66-24
History Note: Statutory Authority G.S. 143-215.3(a) (1);	66.27
143-215.3(a) (10);	66.28
Eff. February 1, 1976;	66.29
Amended Eff. December 1, 1984.	66.30
.0205 CERTIFICATION AND RENEWAL OF CERTIFICATION	66.32
	66.33
(a) Prerequisites for Certification (1) Water. Analytical methods shall conform to those found	66.35
	66.36
52780 (December 1, 1976) including any amendments	
through June 1, 1984, as submitted by the Environmental	
seq, Vol. 41 Federal Register lists methods from the	00.42

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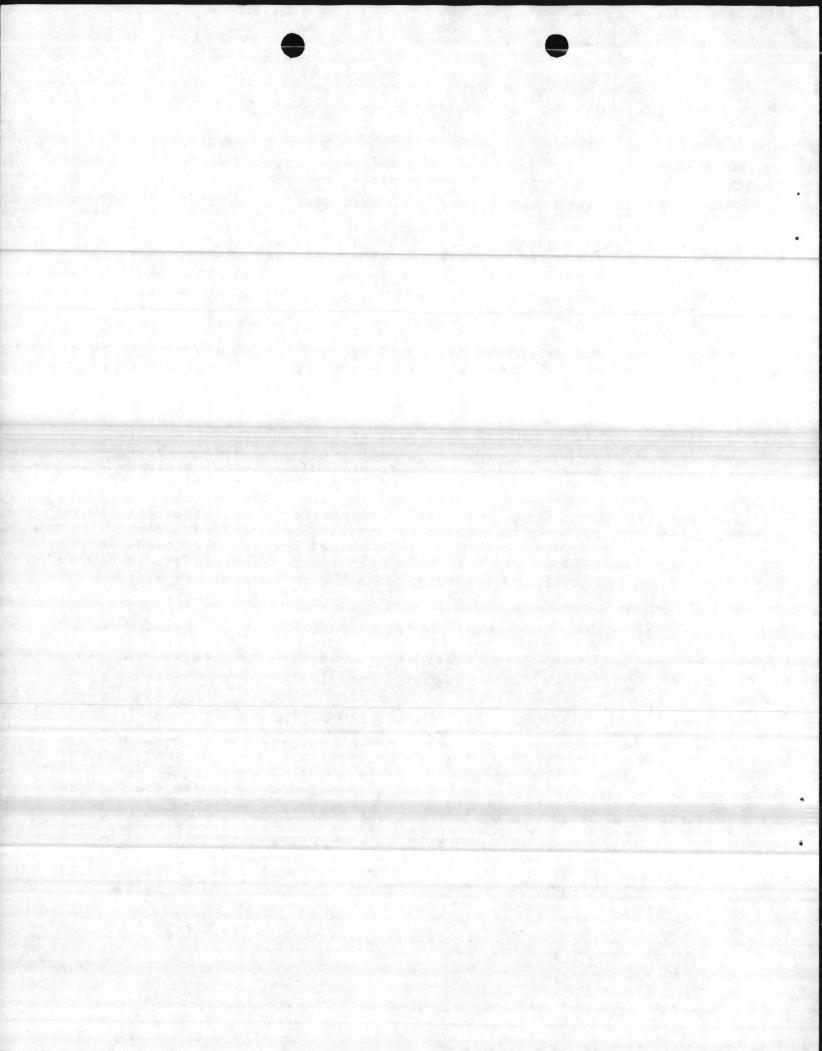
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17)

14th Edition of "Standard Methods for the Examination of Water and Wastewater" (published jointly by the 66.43 66.44 American Public Health Association, the American Water Works Association; and the Water Pollution Control Federation) and "Methods for Chemical Analysis of Water 66.45 and Wastes", 1974 (prepared by the U.S. Environmental 66.46 Protection Agency). The 15th Edition of "Standard 66.47 Sethods for the Examination of Water and Wastewater" and the 1979 "Methods of Chemical Analysis of Water and 66.48 Wastes" have reprinted most of the approved procedures. 66.49 In cases where these references have reprinted the same 66.50 procedures with no changes, they are acceptable for use in compliance with these Regulations. 66.51 laboratory must 66.53 Performance Evaluations. Each demonstrate satisfactory performance on evaluation 66.54 samples submitted by the State Laboratory. 66.56 Supervisory Requirements. The supervisor of a commercial laboratory must 67.2 (3) have a minimum of a B.S. or A.B. degree from an 67.3 accredited college or university in chemistry or closely related science curriculum plus a minimum 67.4 of two years laboratory experience in analytical 67.5 chemistry, or a two year associate degree from an 67.6 college, university, or technical accredited institute in chemistry technology, environmental sciences, or closely related science curriculum 67.7 67. R plus a minimum of four years experience in analytical chemistry. Non-degree supervisors must have at least six years laboratory experience. 67.9 67.11 The supervisor of a municipal or industrial waste (B) water treatment plant laboratory must have a 67.12 minimum of a B.S. or A.B. degree from an accredited college or university in chemistry or 67.13 closely related science curriculum plus a minimum of six months laboratory experience in analytical 67.14 chemistry, or a two year associate degree from an 67.15 university, OL technical accredited college, institute in chemistry technology, environmental 67.16 sciences, or closely related science curriculum 67.17 a minimum of two years experience in plus 67.18 analytical chemistry. Non-degree supervisors must have at least six years laboratory experience. 67.19 67.21 All laboratory supervisors are subject to review (C)by the State Laboratory. One person may serve as supervisor of no more than two laboratories. 67.22 The supervisor shall provide personal and 67.23 direct supervision of the technical personnel and te held

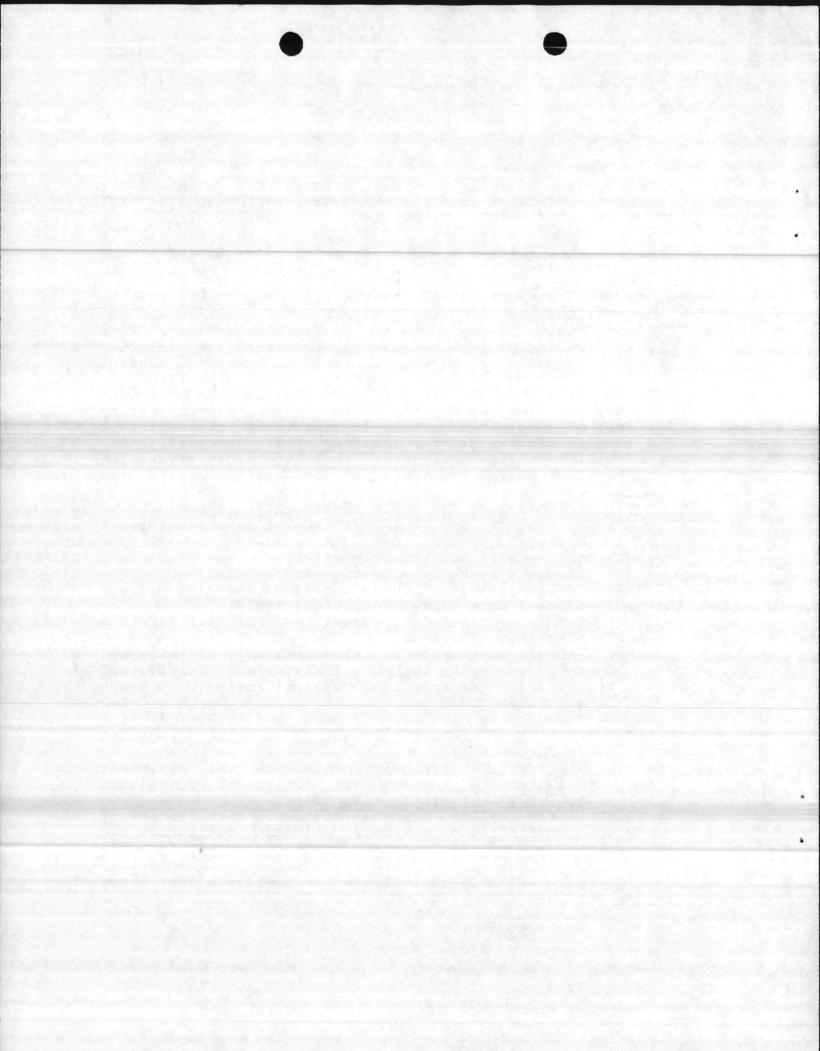
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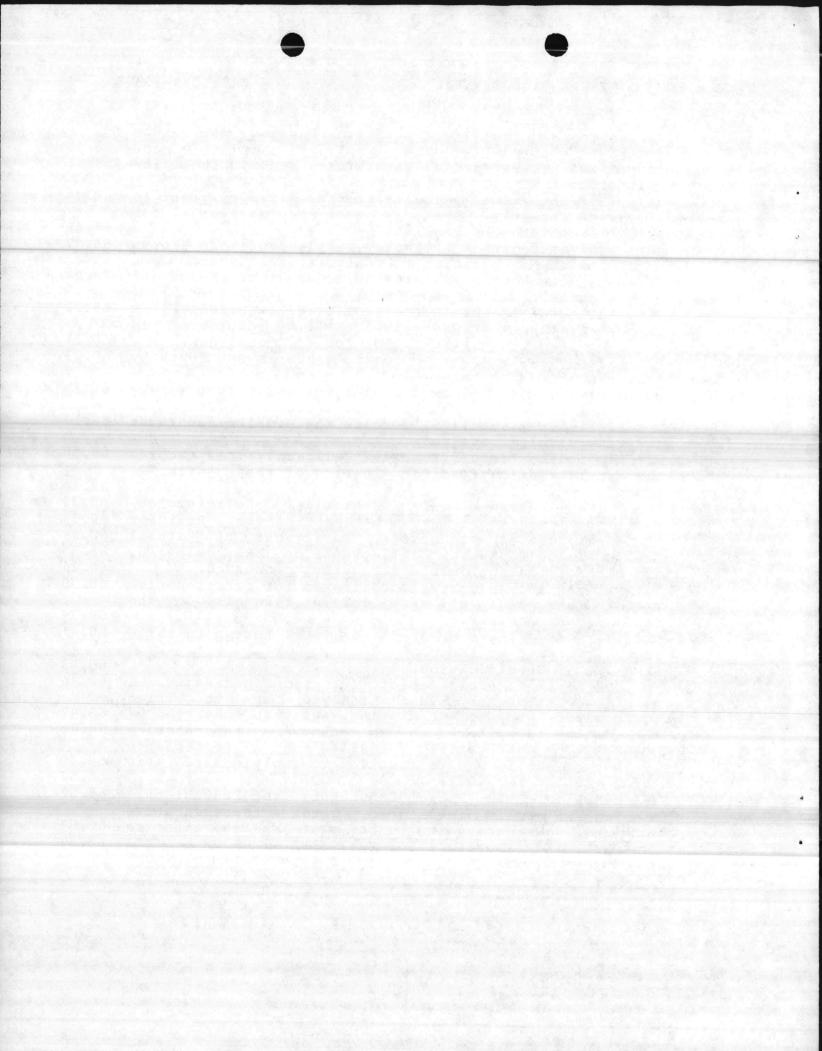
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	responsible for the proper performance and	67.24
	reporting of all analysis made for these	
	Regulations. If the supervisor is to be absent,	67.25
	the supervisor shall arrange for a substitute	
	capable of insuring the proper performance of all	
		67.27
	laboratory procedures. Existing laboratory	
	supervisors that do not meet the requirements in	67.28
	this Paragraph may be accepted after review by the	
	State Laboratory and meeting all other	67.30
	certification requirements.	
(4)	Application. Each laboratory requesting state	67.31
7.41	certification or certification renewal shall submit an	
	application in duplicate to the State Laboratory. Each	
	application will be reviewed to determine the adequacy	67.34
	of personnel, equipment, records, quality control	
	procedures, and methodology. After receiving a	67.35
	completed application and prior to issuing	67.36
	certification, a representative of the State Laboratory	07.50
		17
	may visit each laboratory to verify the information in	
	the application and the adequacy of the laboratory.	67.38
151	Facilities and equipment. Bach laboratory requesting	67.39
	certification must contain or be equipped with the	67.40
	following:	
	(A) A minimum of 150 sq ft of laboratory space;	67.42
	(B) A minimum of 12 linear feet of laboratory bench	
		07.45
	space;	17 111
	(C) A sink with hot and cold water;	67.46
	(D) Adequate lighting, cooling, and heating:	67.48
	(E) An analytical balance capable of weighing 0.1 mg,	67.51
	mounted on a heavy shock proof table;	
	(P) A refrigerator of adequate size that will maintain	67.53
	temperature of 4°C;	
		67.55
	(G) An EPA approved or a current copy of "Standard	07.33
	Methods for the Analysis of Water and Wastewater"	and the second of
	or EPAs "Methods for Chemical Analysis of Water	67.56
	and Wastes";	
	(H) A source of distilled or deionized water that will	68.1
	meet the minimum criteria of the approved	Sec. Sec. Sec.
	methodologies:	
		68.3
	(I) Glassware, chemicals, supplies, and gquipment	00.3
	reguired to perform all analytical procedures	
	included in their certification.	68.4
(61	Analytical Quality Control Program. Each laboratory	68.6
and the	shall develop and maintain a document outlining the	
	analytical quality control practices used for the	68.7
		68.8
	parameters included in their certification. Supporting	
	records shall be maintained as evidence that these	68.9

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practices are being effectively carried out. The quality control program shall be available for 68.10 inspection by the State Laboratory and include the 68.11 following: All analytical quality control data pertinent to 68.14 (A) each certified analysis must be available for 68.15 inspection upon request. Analyze one duplicate sample and one known 68.17 (B) standard in addition to calibration standards each day samples are analyzed to document precision and 68.18 accuracy. Any quality control procedures required by a 68.20 (C) particular approved method shall be considered as required for certification for that analysis. 68.21 All quality control requirements as set forth by 68.23 (D) the State Laboratory. A corrective action policy requiring that at any 68.25 (E) quality control results indicate an time analytical problem, resolve the problem and rerun 68.26 any samples involved. A policy requiring that all analytical records 68.28 (F) must be maintained for a period of three years. 68.30 (b) Issuance of Certification 68.32 deficiencies, substantial absence of (1) In the 68.33 certification will be issued by the Director, Division Environmental Management, Department of Natural 68.34 of Resources and Community Development, or his delegate, 68.35 68.36 for each of the applicable parameters requested. Initial certifications will be issued for prorated time 68.38 (2) periods to schedule all certification renewals on the 68.39 first day of January. 68.41 Initial certification shall be valid for up to three (3) years from date of issue. 68.43 (c) Maintenance of Certification. 68.46 maintain certification for each parameter, a (1) TO certified laboratory must analyze up three to performance gvaluation samples per year submitted by 68.47 68.48 Laboratories the State Laboratory as an unknown. submitting unacceptable results on a performance evaluation sample may be required to analyze more than 68.49 68.50 three samples per year. 68.52 In addition, the State Laboratory may request that (?) samples be split into two equal representative portions, one part going to the state and the other to 68.53 68.54 the certified laboratory for analysis.

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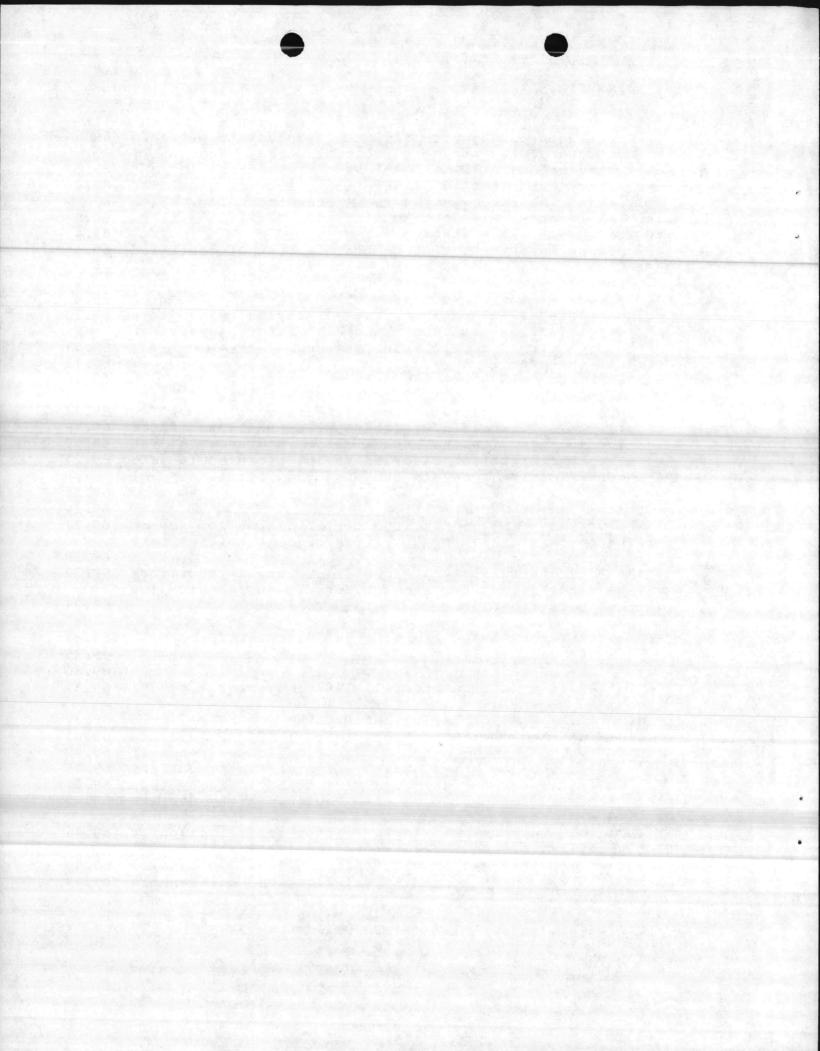


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(3) A certified laboratory will be subject to periodic 6 in spections during the certification period and shall	8.56
	8.57
	9.2
commercial laboratories and the parameters for which	
	9.3
	9.4
	9.6
municipal and industrial laboratories and the	
	9.7
	9.8
	9.10
Laboratory with written notice of laboratory supervisor	
changes within 30 days of such changes. 6	9.11
(d) Certification Renewals. 6	9.13
(1) Applications for certification renewal will be 6	9.16
submitted in duplicate to the State Laboratory 30 days	N. Contract
	9.17
	9.18
	9.19
	9.21
all certification renewals due on the first day of	7.21
	9.22
	9.24
	9.27
all parameters by making a written request to the State	
	9.28
	9.30
may be recertified by meeting the requirements for	100400
initial <u>c</u> ertification. 6	9.31
"istory Note: Statutory Authority G.S. 143-215.3(a) (1); 6	9.34
	9.35
	9.36
	9.37
Amended Eff. December 1, 1984; November 1, 1978. 6	9.31
OPOF THES ASSOCIATED WITH CERTIFICATION PROGRAM 6	9.39
	9.41
	9.42
	9.43
	9.44
	9.45
(\$250.00).	-
	9.46
이 문제 이 지 않는 것 같은 것 같	9.47
이는 그는 것이 같은 것에서 한 것을 수 있는 것이다. 이렇게 잘 많이 잘 많이 가지 않는 것이 없이 가지 않는 것이 없는 것이 같이 않는 것이 않 않는 것이 않는 않는 것이 않 않 않는 것이 않는 않는 것이 않 않 않 않 않 않 않 않 않 않 않 않 않 않 않 않 않 않	9.48
fifty dollars (\$250.00) each year. Certification maintenance 69	9.49

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NRECD - ENVIRONMENTAL MANAGEMENT

fees will not be required for those years in which certification 69.50 or certification reneval are required. These fees are due on or 69.51 before the first day of January or the certification anniversary 69.52 date. • 69.53 (c) Pees may be prorated in order to make all certification 69.54 renewals due on the first day of January. (?) Out-of-state laboratories shall reinhurse the state for 69.55 actual travel and subsistence costs incurred in certification and 69.56 69.57 maintenance of certification. ased and the History Note: Statutory Authority G.S. 143-215.3(a) (1); 70.3 70.4 143-215.3 (a) (10); 70.5 Eff. February 1, 1976; 70.6 Amended Eff. December 1, 1984. 70.8 .0807 DECERTIFICATION (a) Laboratory Decertification. Once certified, a laboratory 70.10 70.11 may lose its certification for all parameters by failing to: 70.14 Maintain the facilities, or records, or personnel, or (1) equipment, or quality control program as set forth in 70.15 the application and these Regulations; or 70.17 (2) Submit truthful and accurate data reports; or 70.18 (3) Pay required fees by the date due; or (4) Discontinue supplying data for clients or programs 70.21 described in Rule .0802 of this Section during periods 70.22 when a parameter decertification is in effect. 70.23 (b) Parameter Decertification. Once certified, a laboratory 70.24 may loose its certification for a a parameter by failing to: (1) Obtain acceptable results on two consecutive 70.27 performance evaluation samples submitted by the State 70.28 Laboratory; or (2) Obtain acceptable results on three consecutive split 70.30 samples that have also been analyzed by the State 70.31 Laboratory; or (?) Submit a split sample to the State Laboratory as 70.33 requested : or 70.34 (4) Use approved methods of analysis; or (5) Report equipment changes that would affect its ability 70.37 to perform the test within 30 days of such changes; or 70.38 (6) Report analysis of performance evaluation samples 70.40 submitted by the State Laboratory within 30 days of 70.41 receipt; or 70.43 [7] Maintain records and perform guality controls as set forth by these Regulations and the State Laboratory for 70.44 a particular parameter; or 70.46 Maintain equipment required for a particular parameter. 70.48 (c) Decertification Requirements.

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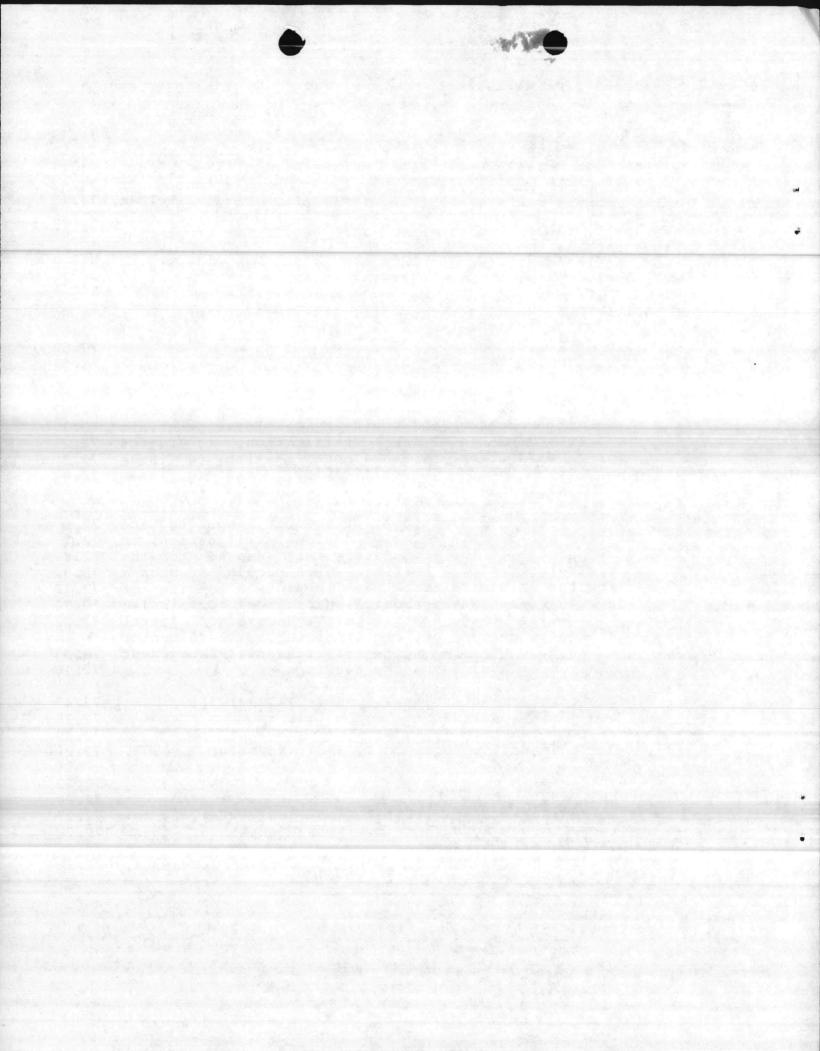
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clients that the analysis cannot be supplied. The 205 period and supply the State Laboratory with the name of the certified laboratory to be used. 71.9 Amended Eff. December 1, 1984. sufficient or adequate facilities, or laboratory supervisor, or gamples similar to those for which approval was lost. Pecertification gamples may be requested at any time, however, div pariod issociately following the fate of decartification. (c) 1 laboratory decartified for falsified reports looses History Woter Statutory Authority G.S. 143-215. 3(a) (1); F TRANSMAN 323

NBSCD - FNVIRONMENTAL MANAGEMENT.

.0809 PECIPROCITY 71.38 (a) Laboratories certified under other state certification 71.40 programs may be given reciprocity certification where such 71.41 programs meet the requirements of these Regulations. In requesting reciprocity certification, laboratories shall include 71.42 71.43 with the application required by Regulation .0805(a) of this 71.44 Section a copy of their certification and Regulation from the 71.45 certifying agency. (b) Laboratories certified on the basis of program equivalency 71.46 shall pay the fees required by Regulation .0806 of this Section. 71.47 History Note: Statutory Authority G.S. 143-215.3(a) (1); 71.50 143-215.3 (a) (10) : 71.51 Eff. February 1, 1976; 71.52 Amended Eff. December 1, 1984. 71.53 . 0910 ADMINISTRATION 71.55 The Director of the Division of Environmental Management, (a) 71.57 Department of Natural Resources and Community Development, or his 72.1 de'egate, is authorized to issue certification, to 72.2 reject oplications for certification, to renew certification, to issue 72.3 recertification, to issue decertification, and to issue 72.4 reciprocity certification. Appeals. In any case where the Director of the Division (b) 72.5 of Environmental Management, Department of Natural Resources and 72.6 Community Development or his delegate denies certification, or 72.7 decertifies a laboratory, the laboratory may appeal to the director or his delegate for a hearing. Upon receipt of such a 72.8 72.9 request, the director or his delegate shall convene a hearing of 72.10 the type provided for Environmental Management Commission 72.11 Pegulation in 15 NCAC 2I .0300, Administrative Hearings. 72.12 Appeal from the decision of a hearing officer appointed by the director or his delegate shall be governed by the Environmental Management 72.13 Commission Pegulation on administrative hearings. 72.14 History Note: Statutory Authority G.S. 143-215.3(a) (1); 72.17 143-215.3(a) (10); 72.18 Eff. February 1, 1976; 72.19 Amended Eff. December 1, 1984; November 1, 1978. 72.20 .0911 IMPLEMENTATION 72.22 (3) Commercial Laboratories 72.24 Certified commercial laboratories must meet any new (1) 72.27 requirements set forth herein within 6 months of these 72.28 Regulations effective date. Certification fee changes are effective January 1, (2)72.30 1985.

NOPTH CAROLINA ADMINISTRATIVE CODE 11/20/84



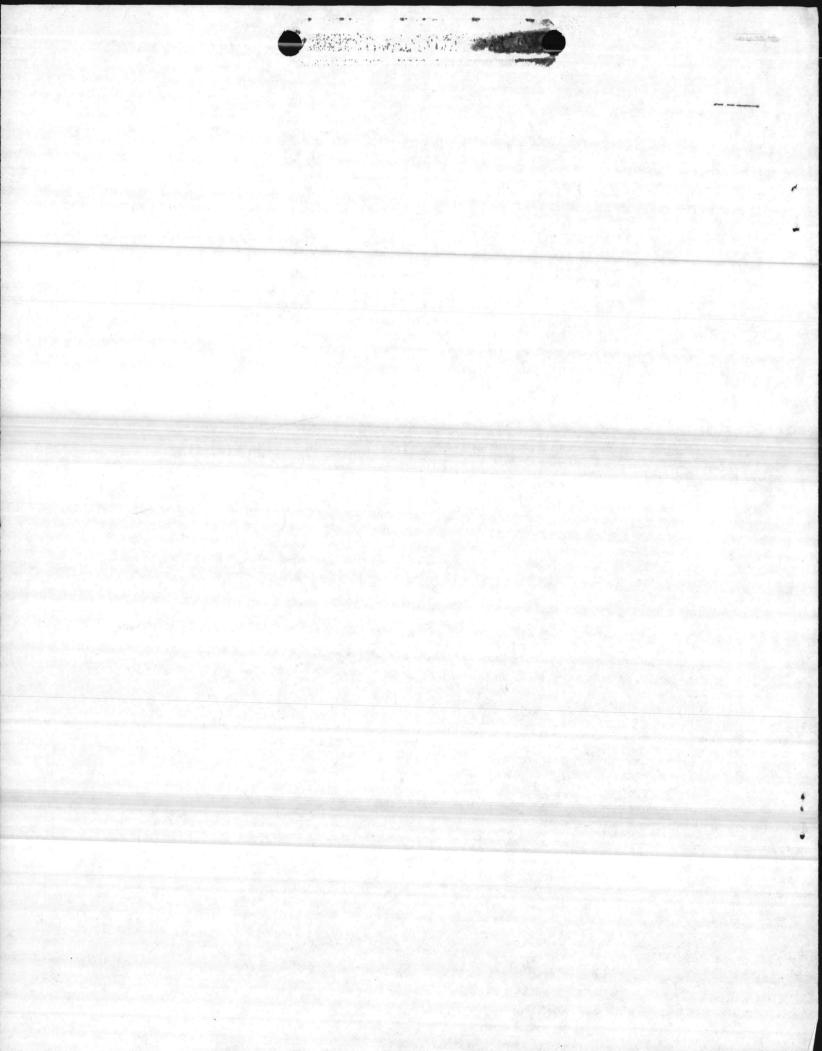


NRECD - ENVIRONMENTAL MANAGEMENT

- (3) Requests for new parameters can be made by submitting a 72.32 proper application form.
 (b) Municipal and Industrial Laboratories 72.33
 - (1) All Municipals and Industrial Waste Treatment Plant 72.36 Laboratories subject to Rule .0802 of these Regulations 72.37 are required to be certified.
 - All Municipal and Industrial Waste Treatment Plant 72.39 (2) 72.40 Laboratories subject to Rule .0802 of these Regulations nust submit an application for certification within six of months these Regulations effective date. 72.41 Laboratories submitting an acceptable application will 72.42 be considered in compliance with these Regulations 72.43 until the State Laboratory can process the application 72.44 and issue or deny certification.
 - (3) Laboratories that cannot meet initial certification 72.46 requirements must comply with the Decertification Requirements as set forth in Rule .0807(c) of these 72.47 Regulations.

History	Note:	Statutory Authority G.S. 143-215.3(a) (1);	72.50
		143-215.3 (a) (10);	72.51
		Eff. December 1, 1984.	72.52

NOPTH CAPOTINA ADMINISTRATIVE CODE 11/20/84



T- 5221/3



State of North Carolina Department of Natural Resources and Community Development 512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor

S. Thomas Rhodes, Secretary

Division of Environmental Management

APR 5 1985

Dear NPDES Permit Administrator:

RE: Quality Assurance Guidance and Steps Involved in Securing Certification

Enclosed is the quality assurance guidance that was promised in Mr. Helms' January 1985 letter that transmitted wastewater laboratory certification information. Part .0805(a)(6) of the certification regulation states that each laboratory shall develop and maintain a quality assurance document outlining the quality control practices of the laboratory. Laboratories are not expected to have a quality control document, but are expected to begin developing one.

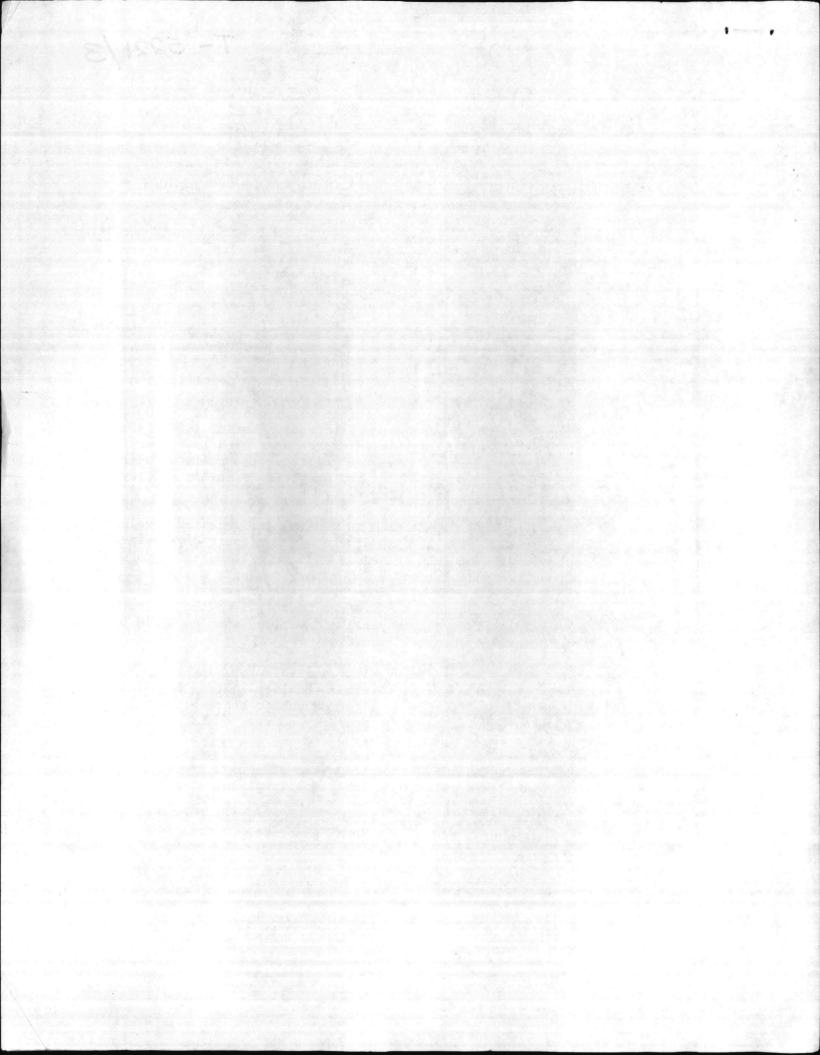
Parts I - III of the enclosed information is general guidance for developing a quality control plan and part IV is a listing of the minimum controls required for certification. Although it will be July or August 1985 before we begin processing certification applications, we encourage each laboratory to begin implementing the required quality controls so they will be better prepared for certification. We will consider substituting existing programs that are not identical to the enclosed required program. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

Questions have been raised concerning quality controls for parameters for which standards are not readily available (Ex. Coliform). Quality control requirements for these parameters have been modified. At a future public hearing, we will propose changes in the certification regulations that properly address quality controls for these parameters.

Some laboratories have requested information about the steps involved in securing certification. The following is a summary of the steps involved.

- 1. The laboratory completes the application forms and submits them to the Division of Environmental Management (DEM) Laboratory.
- 2. DEM Laboratory reviews the applications, notes any deficiencies and returns the application for any needed information.

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-4984



NPDES Permit Administrator Page 2

- 3. DEM Laboratory mails performance evaluation samples to the laboratories seeking certification. This will begin in July or August 1985.
- 4. Your laboratory analyzes the samples and submits results to the DEM Laboratory.
- 5. DEM Laboratory reviews and evaluates these results and, if necessary, sends rerun samples. The DEM Laboratory will assist any laboratories having problems producing acceptable results. Assistance may be in the form of known samples, methodology, work forms, or quality controls.
- 6. The DEM Laboratory schedules and performs an onsite laboratory inspection. Time sensitive samples may be delivered during the inspection.
- 7. An inspection report listing any observed deficiencies will be prepared and mailed.
- 8. The laboratory seeking certification agrees to take any required corrective actions. Reasonable time will be given for taking corrective actions.
- 9. The laboratory requesting certification is billed the appropriate fees.
- 10. Upon receipt of payment, DEM issues certification for up to three years.

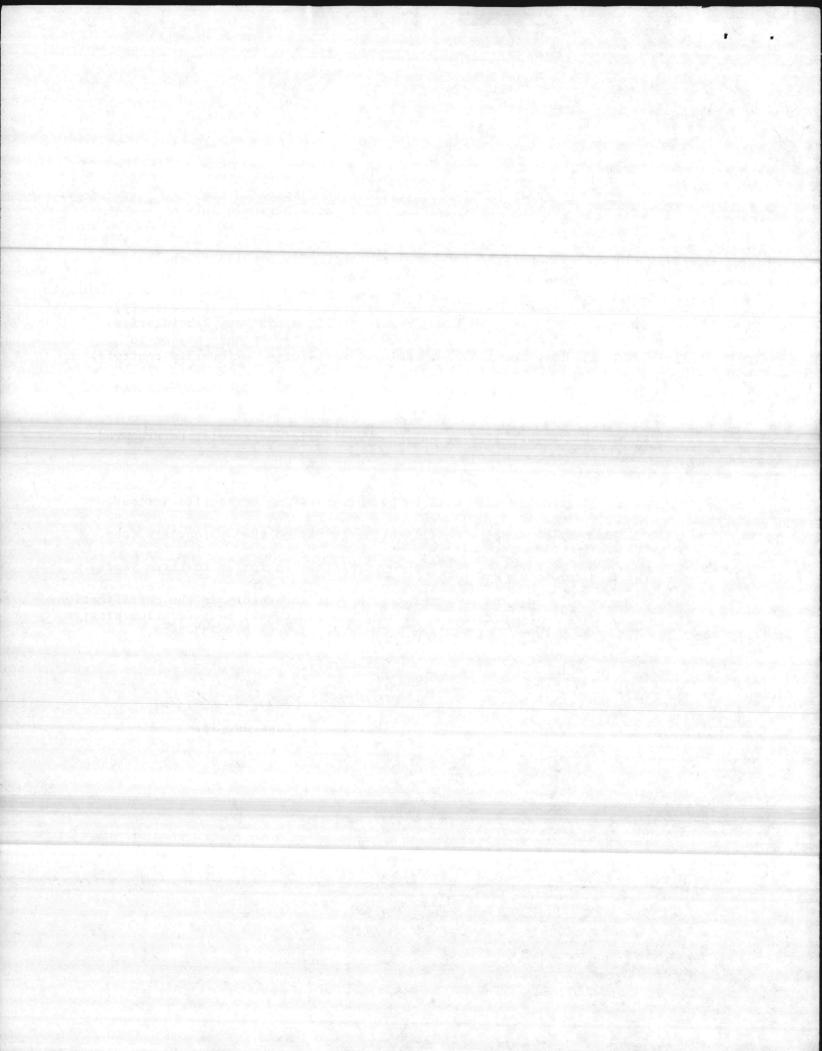
We continue to look forward to working with your laboratory in the certification program. If you have any questions concerning the laboratory certification program, contact Mr. Billy D. Byrd or Mr. William B. Edwards, Jr. at 919-733-3908.

Sincerely,

W. B. Edwards, gs.

William B. Edwards, Jr.

Enclosure



NRCD/DEM Wastewater Laboratory Certification Guidance for Preparing a Quality Assurance Document

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I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures. 5 TO

II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

General Laboratory Practices III.

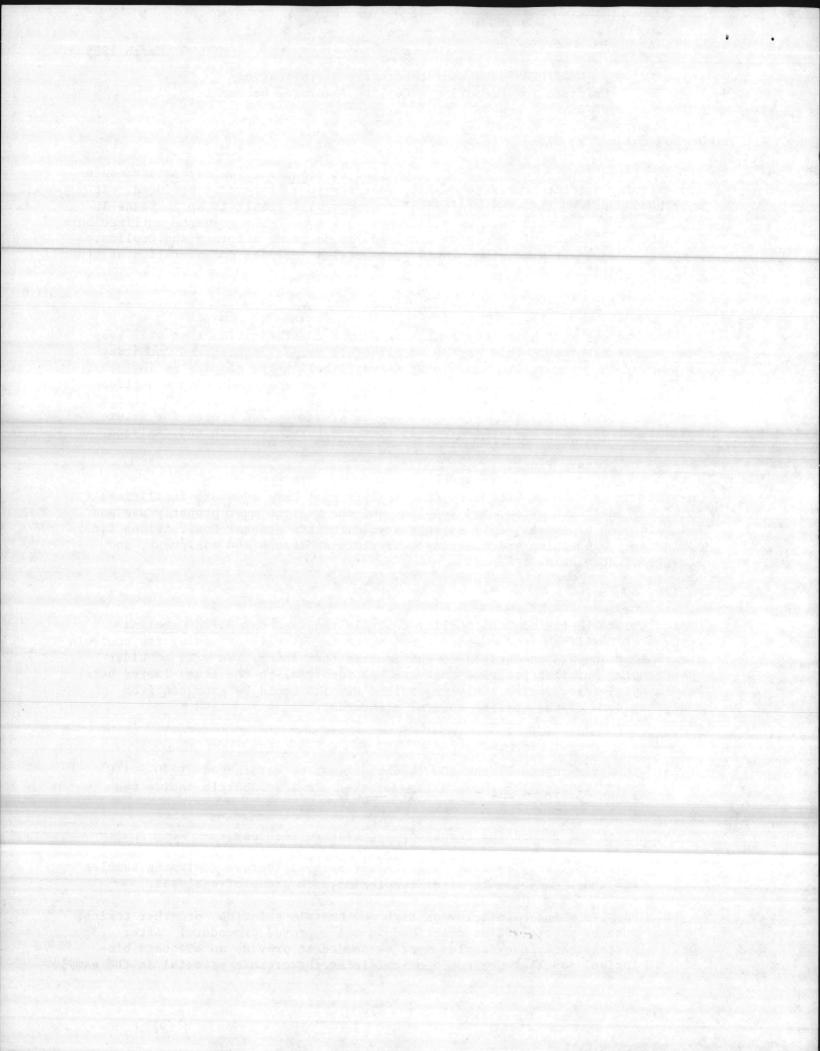
In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals. COD schodard

IV. Quality Controls svoda and to a 0.01 attuin

Listed below are the minimum quality controls required for North Carolina Wastewater Certification. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

1. BOD histats sit it four set of symplet cards her deald a lo behands that when

- a. The temperature of the BOD incubator must be maintained at 20 \pm 1°C using an accurate thermometer inserted in a BOD bottle inside the incubator.
- Check and record the incubator temperature each day. Ъ.
- c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses (USE BLANK)
- Samples which have a low or high pH, contain chlorine, or other toxics, d. must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.



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- e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.
- f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/1.
- g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.
- h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.
- 1. Analyze a duplicate sample daily.
- j. Perform a blank dilution water control analysis along with each batch of samples analyzed.
- 2. COD Titration Procedure selected and the selected and the state
 - a. Standardize the COD titrant each day samples are analyzed.
 - b. Analyze a distilled water blank with each group of samples and make proper corrections.
 - c. Analyze a quality control standard along with each group of samples analyzed.

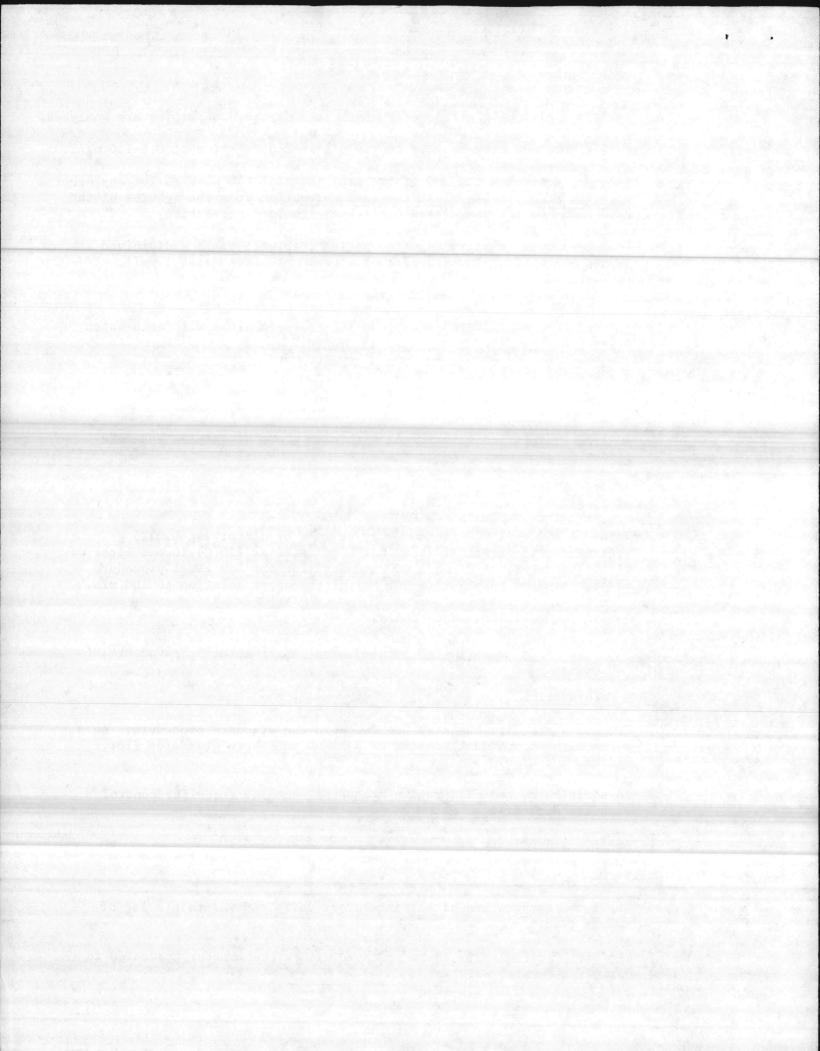
Note: A 250 mg/1 COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/1 COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

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- d. Analyze a duplicate sample daily.
- e. Use the low level procedure for the analysis of samples with a COD of <50 mg/1.

3. <u>COD</u> - <u>Colormetric</u>, abcabase is the galaxies to the solution of the solu

- a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).
- b. In addition to the calibration standards, analyze a quality control standard each day.
- c. Analyze a duplicate sample daily.
- 4. Collication the dissolved mater sach day belows and the distriction at the distribution of the distribu
 - a. Check the temperature of all incubators daily and maintain a log of values read,
 - b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.



- c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.
- d. Log the maximum temperature and pressure of the autoclave once during each use.
- e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.

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- f. Analyze one duplicate sample each day.
- 5. Chloride
 - a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.

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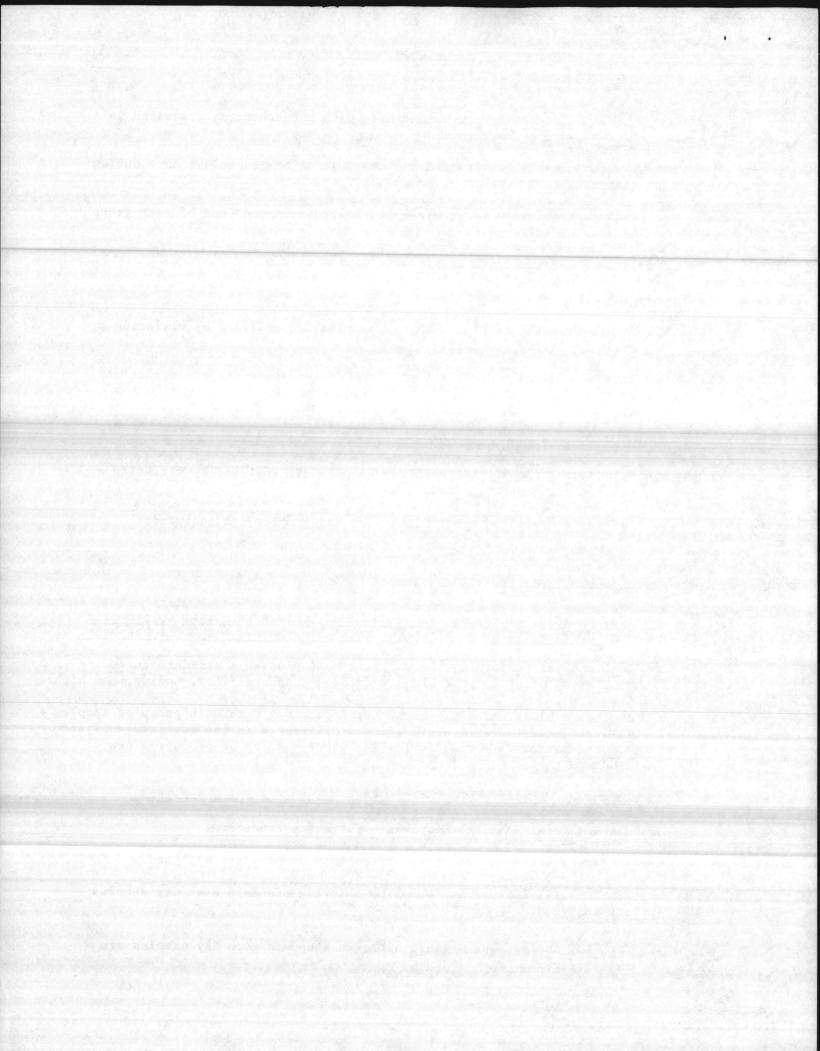
- b. Analyze a distilled water blank each day and make proper corrections.
- c. Analyze one duplicate sample each day.
- 6. Hardness
 - a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.
 - b. Analyze a distilled water blank each day samples are analyzed.

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- c. Analyze one duplicate sample daily.
- 7. Colormetric Analyses: Diabase for

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

- a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).
- b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.
- c. Analyze a duplicate sample with each group of samples analyzed.
- 8. Ammonia and Total Kjeldahl Nitrogen Titration Procedure
 - a. Analyze a distilled water blank each day samples are analyzed.
 - b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.
 - c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.



Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing l ml concentrated H_2SO_4 and diluting to one liter. Diluting 10 ml of this standard and l ml concentrated H_2SO_4 to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

d. Analyze a duplicate sample daily.

9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

- a. Calibrate the meter according to the manufacturer's instructions.
- b. Check the meter calibration by analyzing a medium level quality control standard each day.

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c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

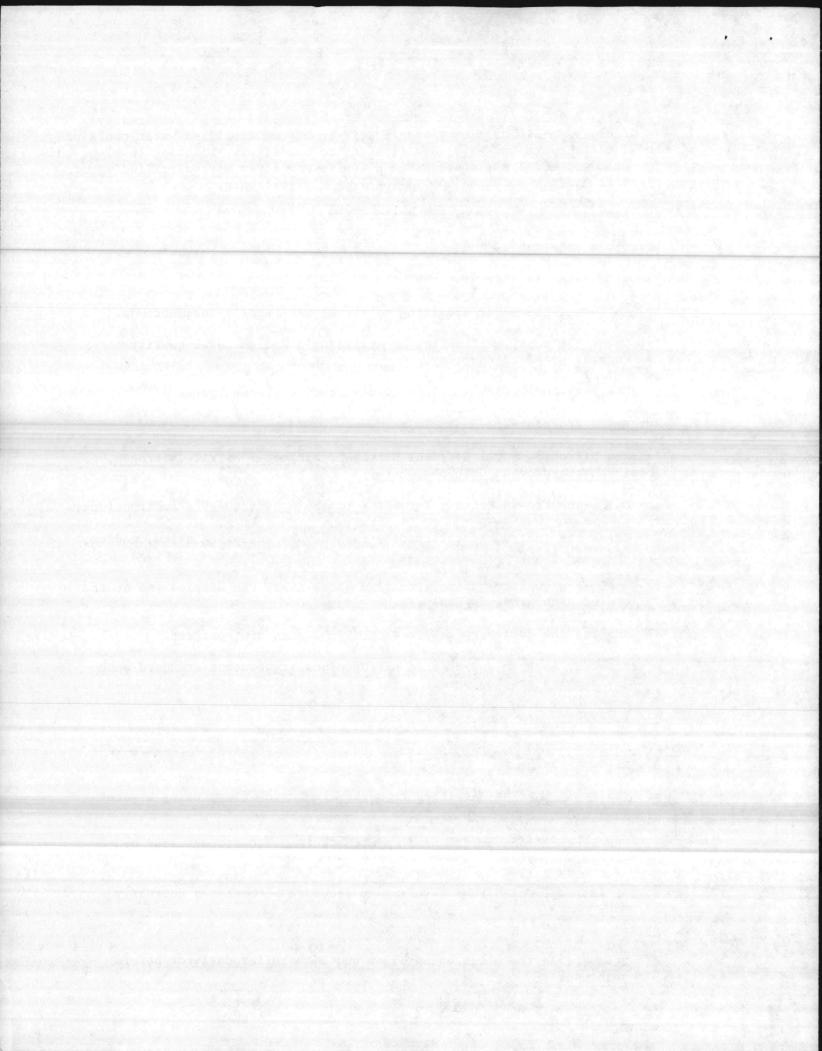
Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

- a. Calibrate the instrument according to the manufacturer's instructions.
- b. Check the instrument calibration each day by analyzing a low, medium, and high standard.
- c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.
- d. Analyze one duplicate sample each day samples are analyzed.
- e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

- a. Perform a blank analysis on each batch of freon used and make proper corrections.
- b. The freen must be distilled from the extraction flask using a water bath controlled at 70°C.
- c. The extract must be filtered through Whatman #40 filter paper or equivalent.
- d. It is recommended that a reference standard be analyzed quarterly.
- 12. pH
 - a. Standardize the meter using a low and high buffer daily or before each use.

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- b. It is recommended that a reference standard be analyzed quarterly.
- c. Analyze a duplicate sample daily.
- 13. Total Residue and Total Suspended Residue
 - a. Check and record drying oven temperature each day used.

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- b. Analyze one duplicate sample each day samples are analyzed.
- c. It is <u>recommended</u> that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.
- d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

- a. Standards as described in the approved procedure must be secured and used.
- b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.
- c. Analyze one duplicate sample each day samples are analyzed.
- d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

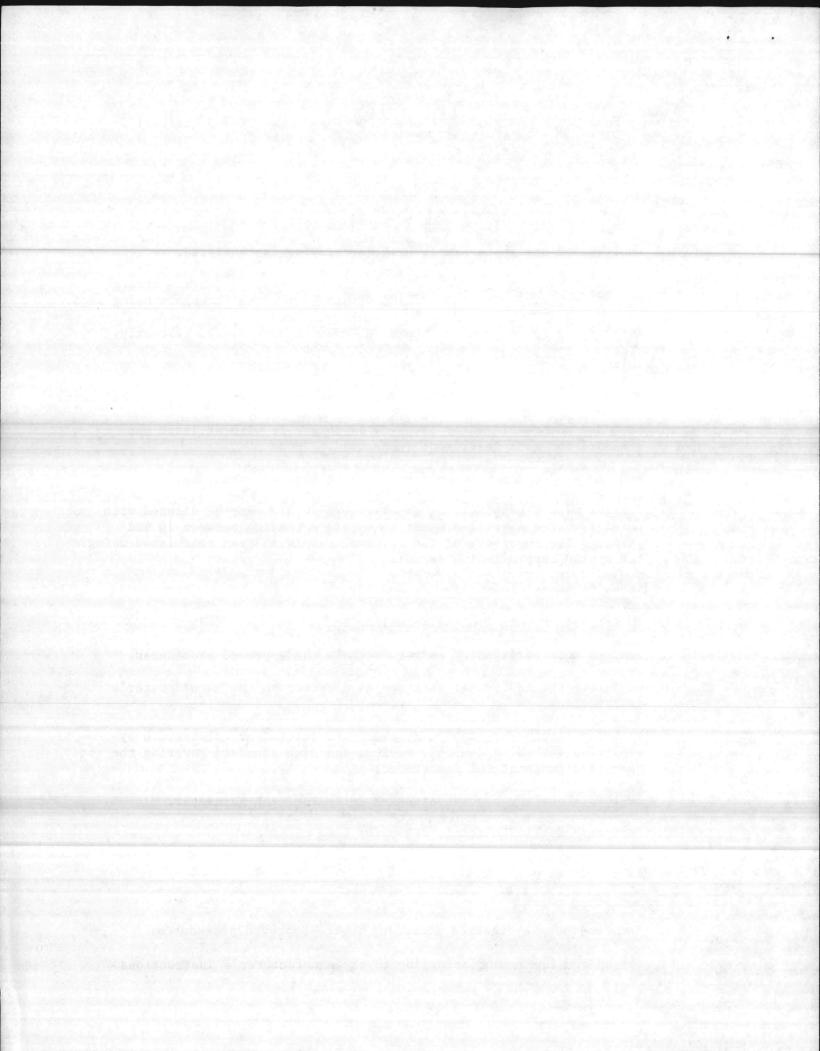
- a. Samples must be digested as set forth in the approved procedures.
- b. Calibrate the instrument each day as directed in the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily for each parameter.
- e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

- a. Samples must be digested according to the approved procedures.
- b. Set up the instrument according to the manufacturer's instructions.

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- c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.
- d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.
- e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

- a. Samples must be digested according to the approved procedures.
- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.
- c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.
- d. Analyze one duplicate sample daily.
- 18. Mercury
 - a. Set up the instrument according to the manufacturer's instructions and the approved procedure.

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- b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.
- c. In addition to the calibration standards, analyze a quality control standard daily.

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d. Analyze one duplicate sample each day samples are analyzed.

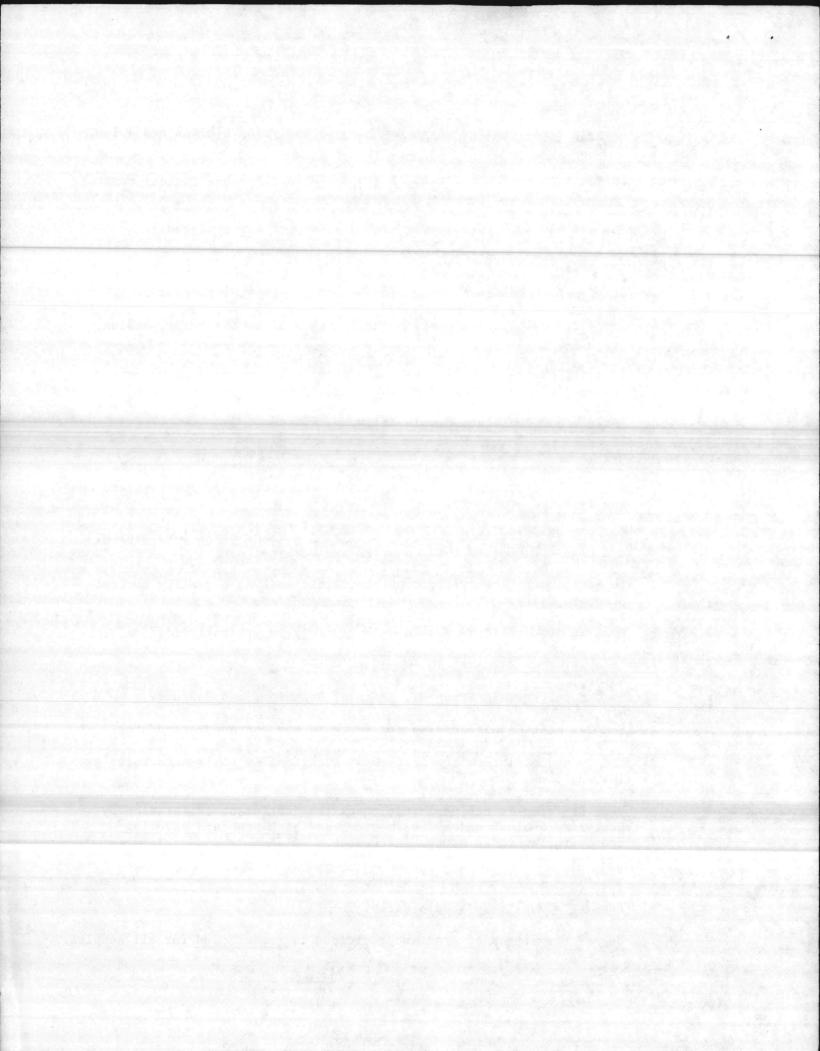
19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

- a. Samples must be digested as set forth in the approved procedures.
- b. Set up and calibrate the instrument according to the manufacturer's instructions.
- c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.
- d. Analyze a quality control standard daily.
- e. Analyze a duplicate sample daily.
- f. Use of the method of standard additions is recommended for all samples.

16. Metals Hydraids

-6-



20. Analytical Balance

a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.

-7-

- b. Weigh a standard weight each day the balance is used and maintain a log of values read.
- c. Check the balance zero before each use.
- d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

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21. Approved Procedures

- a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.
- b. A copy of the approved reference procedures must be available in the laboratory.

22. Chemicals, Reagents, and Glassware

- a. Reagents must be prepared and used as detailed in the reference procedures.
- b. Date all chemicals received and all reagent solutions prepared.
- c. All chemicals should be reagent grade, when available.
- d. Maintain a record of all standardizations performed.
- e. We recommend that all glassware be Class A, when available.

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23. Sample Preservation

- a. Samples should be preserved immediately after collection.
- b. Document the type of preservatives that are to be used and when samples are preserved.

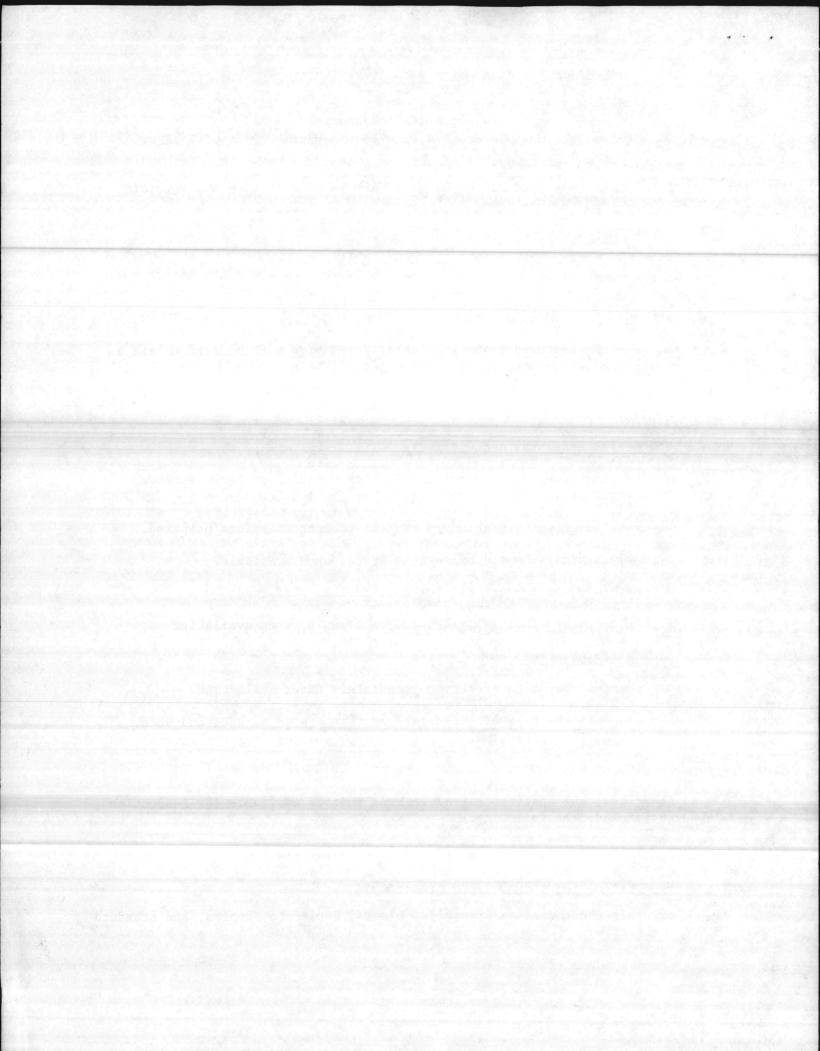
24. Records

Analytical and quality control records must be available for inspection and include the following:

a. Date samples are collected and date analyzed.

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- b. Daily lab worksheets and workbooks.
- c. Values obtained on standards, blanks, duplicate samples, and standard curves.



- d. A record of all required quality controls.
- e. All worksheets must contain the signature or initials of the analyst(s) performing that function.

-8-

f. All analytical records must be retained for at least three years.

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25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed \pm 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

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It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

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a. Precisions Control Limits:

Using 30-40 sets of duplicate sample results or an annual data set, calculate precision control limits using the formulas given below:

lange (R)		lst	anal	ysis	- 2nd	analy	sis	
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UCL_R = Upper Control Limit

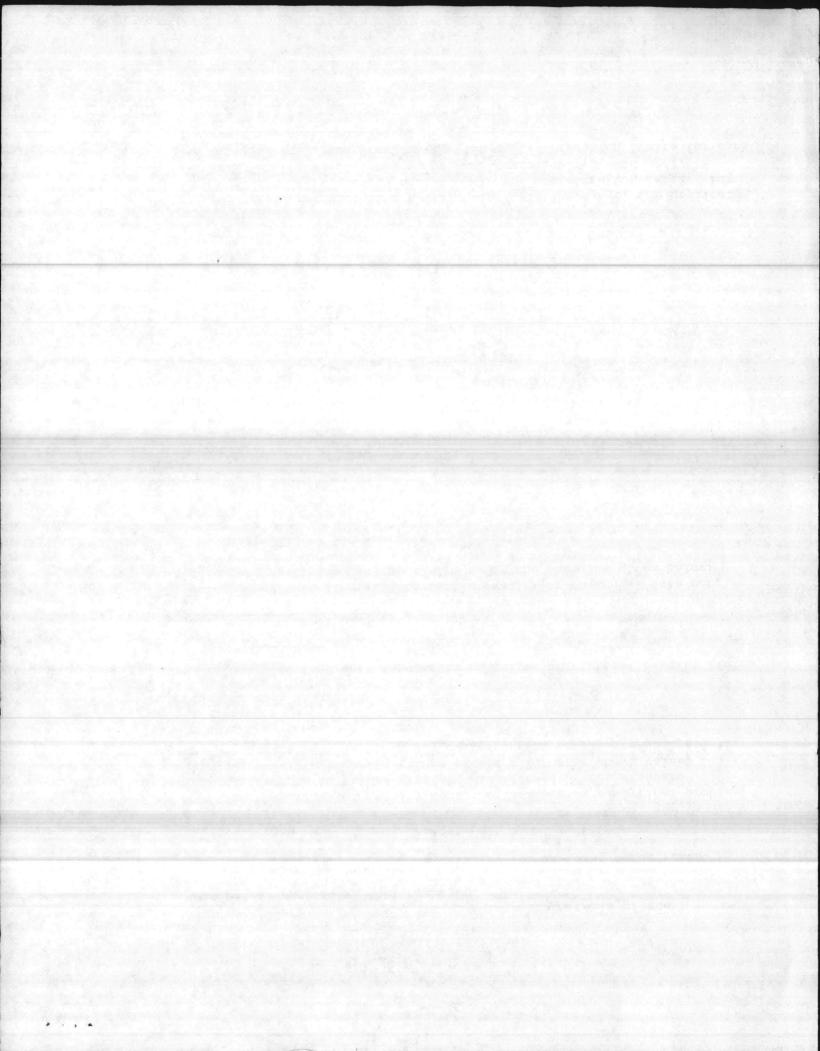
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2.51 = Shewhart factor for 2s (duplicate)

Listense List

3.27 = Shewhart factor for 3s (duplicate)

NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1, 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.



b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

P	-	observed	x 1	00						
		known				UCL	=	P +	3 8	Sp
_					$\int \Sigma P^2 - (\Sigma P)^2 = \frac{(\Sigma P)^2}{n}$	UCL p UWL p	-	P +	2 8	Sp
P	-	<u>EP</u> n		Sp-	V	LCLp	-	P -	3 5	Sp
						LWLp	-	P -	2 5	Sp
Wh	ere	: <u>P</u>		Percen	t recovery					

P	-	Percent recovery
P	-	Average percent recovery
n	-	Number of analysis
Sp	-	Standard deviation of percent recovery
UCLp	-	Upper Control limit for percent recovery
UWLp	-	Upper warning limit for percent recovery
LCLp	-	
LWLp	-	Lower warning limit for percent recovery
1000000000	1.112	

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

- (1) These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.
- (2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.
- (3) If seven successive points fall on the same side of the P (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.
- e. For further information concerning statistical quality control limits, we recommend securing a copy of <u>EPA Handbook for Analytical Quality</u> <u>Control in Water and Wastewater Laboratories</u> EPA-600/4-79-019. This can be obtained by writing: Mr. Wade Knight Quality Assurance Officer U.S. EPA, Region 4 College Station Rd. Athens, GA 30613

