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INTRODUCTION

Scope of Analytical Services

This plan describes the North Dakota Department of Health Chemistry Division quality assurance program. The Chemistry Division provides analytical and other support to the entire State Department of Health, with the majority of the analytical support being to the Environmental Health Section. The Chemistry Division also provides analytical support to public water and wastewater systems, the North Dakota Department of Agriculture, the Public Service Commission, the United States Geological Survey, and the State Water Commission. The Chemistry Division also analyzes water samples for private citizens.

Purpose of Quality Assurance Program

The purpose of the quality assurance program is to achieve a level of data quality that meets user requirements for completeness, precision, accuracy, representativeness, comparability, and dependability. The purpose of this written quality assurance program plan is to delineate and document the Chemistry Division's quality assurance practices.

Quality Assurance Policy

It is the policy of the Chemistry Division to conduct appropriate and sufficient quality control checks that enable analytical results to be generated with known precision and accuracy. By doing this, the division's analytical results should be scientifically valid, and defensible. Environmental results must meet quality assurance requirements set by the user. Since quality assurance must be considered an integral and essential part of the overhead associated with generating environmental monitoring data, the amount of data generated by this laboratory will be reduced if adequate manpower is not available to execute the required quality assurance functions.

Manual Format, Revisions, and Distribution

This manual is to be maintained in a loose-leaf notebook to facilitate revisions which are done approximately on an annual basis. Distribution is handled by request.

SECTION I

LABORATORY ORGANIZATION AND RESPONSIBILITY

Organizational Chart

The organizational chart at the end of this section outlines laboratory personnel organization and lines of responsibility.

The ultimate responsibility for the entire quality assurance program rests with the Director, Division of Chemistry. The Director has overall responsibility for the development, implementation, and continued operation of the quality assurance program. Reviewing overall quality assurance records and the ultimate decisions on deleting data or issuing data recalls is the responsibility of the Director after consultation with the appropriate individuals. In addition, if the sample volume is insufficient or if some other factor prohibits a rerun of samples when an out-of-control situation occurs, the ultimate responsibility of editing, flagging, or recalling data lies with the Director. The Director coordinates review, revision, and updating of the quality assurance plan with the quality assurance coordinator. The Director is also responsible for identifying needs for equipment, personnel, and training and for meeting these needs through appropriate coordination with State and EPA personnel, if necessary. Records of continuing education and training are maintained on file for all permanent laboratory personnel. Resumes that indicate educational background and experience are included in these files. Temporary personnel may be on staff at any time. Records of their educational background and experience are maintained on file.

The quality assurance coordinator is organizationally independent of the data generating groups. The quality assurance coordinator works with the Director, Division of Chemistry to review, revise, and update the quality assurance program as necessary. The quality assurance coordinator is responsible for coordinating formal performance evaluation studies and check sample programs which includes ordering the appropriate performance test samples where necessary, preparing the test samples for logging in to the laboratory information management system, completing and sending in results reports, reviewing and distributing returned statistical reports, initiating and following up on analyst's reviews and responses to not-acceptable results, and maintaining associated records. The quality assurance coordinator is also responsible for quality assurance record keeping and evaluating overall laboratory performance for the Chemistry Division.

Analysts are responsible for performing and evaluating the results of quality control checks specified in methodology or EPA requirements. Quality control checks in addition to the minimum required can be performed. Analysts must review the results of quality control checks as soon as possible to determine if an out-of-control situation exists. Analysts should be the first ones to discover a situation that is out of control. An analyte system is out of

control when one quality control result exceeds the control limits (usually the mean plus or minus three standard deviations) or when two consecutive quality control results exceed the warning limits (usually the mean plus or minus two standard deviations), or when seven consecutive quality control results are either all above or all below the mean. When an analytical system is determined to be out of control, the analyst should take appropriate corrective action as outlined in Section XII.

Analysts are responsible for entering quality control results into the LIMS control charting system and for periodically printing control charts. Analysts should review control charts on a regular basis for out of control situations or for trends that indicate potential analytical problems. Analysts also have a part in the data review and verification process.

SECTION II
PROCESS USED TO IDENTIFY
CLIENT'S DATA QUALITY OBJECTIVES

The clients of the Chemistry Division laboratory are varied and include the various environmental monitoring programs in the Water Quality, Municipal Facilities, Waste Management and Air Quality Divisions of the Health Department; municipal drinking water and/or wastewater facilities throughout the state; industrial wastewater facilities throughout the state; the State Department of Agriculture; the Public Service Commission; the United States Geological Survey; the State Water Commission; private citizens, etc.

Data quality objectives used are those in the analytical methodology unless otherwise specified by the client(s). In certain cases managers of the environmental programs meet with the Director, Division of Chemistry and lead analytical personnel to set data quality objectives.

SECTION III STANDARD OPERATING PROCEDURES

The laboratory has two types of written standard operating procedures (SOPs), technical, and general. Technical SOPs describe analytical procedures, and general SOPs describe operations other than analytical procedures. Access to both types of SOPs is available via shared directories on the computer network or via the Internet. The quality assurance coordinator maintains a hard copy file of each SOP. Efforts are made to review the SOPs on a regular basis and make revisions as necessary. Lists of available SOPs can be found in the Tables of Contents for the analytical methods SOPs and the general SOPs.

SECTION IV FIELD SAMPLING PROCEDURES

Sample collectors and sampling locations are not determined by the laboratory. Field sampling personnel coordinate with the laboratory to help smooth the flow of samples to the laboratory. Sampling personnel are requested to give advance notice of sampling trips and expected sample loads. The Director, Division of Chemistry recommends rescheduling of sampling trips when the sample load would surpass laboratory capability.

Sampling procedures, required preservation, proper containers, correct sample container cleaning procedures, sample holding times from collection to analysis, and sample shipping and storage conditions are determined either from the methods or from sources such as *Standard Methods for the Examination of Water and Wastewater*. An individual at the laboratory prepares sample containers (of the proper type and containing the proper preservatives where necessary and applicable) for the individuals who collect samples for analysis of organic contaminants. Sample containers are also provided for collection of samples for analysis of inorganic constituents.

When samples arrive at the laboratory they are checked by sample receiving/log-in personnel for proper containers and temperature. Sample receiving/log in personnel review sample tracking forms for completeness and legibility of entered information. Appropriate analysts check samples for proper preservation prior to analysis, when possible, or after analysis if unable to do so beforehand (e.g. acidification of samples for analysis of volatile organic compounds).

SECTION V LABORATORY SAMPLE HANDLING PROCEDURES

Samples are tracked through the laboratory via a laboratory information management system (LIMS). Each sample is given a unique identification number when it arrives at the laboratory. The identification number is entered into the LIMS along with all pertinent information regarding the sample.

Samples requiring refrigerated storage are stored in a walk-in cooler or in refrigerators.

A chain of custody procedure is used for samples likely to be the basis for an enforcement action.

Policy for rejection of samples:

The following minimum criteria have to be met in order for this laboratory to accept a sample for analysis:

The sample must be properly collected. This includes, but is not limited to, adequate sample size, proper sample container and preservation, if required. Generally, common plastic containers such as milk cartons and soda bottles are unsuitable for sample collection because they may contain components that can interfere with analyte detection (especially for organic analyses). Adequate identification must accompany the sample to provide for the capability of sample tracking.

While not all inclusive, the following table contains much of the pertinent information regarding sample size, container type and preservative on the majority of requested analytes and may be used to determine whether or not to accept a sample. If a parameter is not contained in the table the acceptance or rejection of the sample shall be determined by either the Director of the Chemistry Division or the Assistant Director of the Chemistry Division.

HOLDING TIMES and PRESERVATIVES

MEASUREMENT	mL	OUR CONTAINER	TYPE ^h	PRESERVATIVE	H TIME	ACID VOL ^s
PHYSICAL PROPERTIES:						
Conductivity	100 ^a	Quart Cube	P,G	Cool, 4°C	28 Days	
pH	25 ^a	Quart Cube	P,G	None Req.	0 Hrs.	
TSS	1000	Quart Cube	P,G	Cool, 4°C	7 Days	
Settleable Matter	1000	Gallon Cube	P,G	Cool, 4°C	48 Hrs.	
Temperature	1000 ^a	Quart Cube	P,G	None Req.	0 Hrs.	
Turbidity	100 ^a	Quart Cube	P,G	Cool, 4°C	48 Hrs.	
<u>METALS:</u>						
Dissolved	200	200 mL Bottle	P,G	Filt, HNO ₃ pH<2	6 Mos.	2 mL
Total	200	200 mL Bottle	P,G	HNO ₃ pH<2	6 Mos.	2 mL
Chromium ⁺⁶	1000	Quart Cube	P,G	Cool, 4°C	24 Hrs.	
Mercury, Dissolved	1000	Quart Cube	P,G	Filt, HNO ₃ pH<2	28 Days	4 mL

Mercury, Total	1000	Quart Cube	P,G	HNO ₃ pH<2	28 Days	4 mL
<u>NONMETALS:</u>						
Alkalinity	200 ^a	Quart Cube	P,G	Cool, 4°C	14 Days	
Chloride	50 ^a	Quart Cube	P,G	None Req.	28 Days	
Fluoride	100 ^a	Quart Cube	P,G	None Req.	28 Days	
<u>Nitrogen</u>						
Ammonia	200 ^{b,f}	200 mL Bottle	P,G	4°C, H ₂ SO ₄ pH<2	28 Days	2 mL
Kjeldahl	200 ^{c,f}	200 mL Bottle	P,G	4°C, H ₂ SO ₄ pH<2	28 Days	2 mL
Nitrate/Nitrite	200 ^{c,f}	200 mL Bottle	P,G	4°C, H ₂ SO ₄ pH<2	28 Days	2 mL
Nitrate	200 ^d	200 mL Bottle	P,G	Cool, 4°C	48 Hrs.	
Nitrite	200 ^d	200 mL Bottle	P,G	Cool, 4°C	48 Hrs.	
<u>Phosphorus:</u>						
Orthophosphate, Dis	200	200 mL Bottle	P,G	Filt, 4°C	48 Hrs.	
Total	200 ^{b,f}	200 mL Bottle	P,G	4°C, H ₂ SO ₄ pH<2	28 Days	2 mL
Silica	200	200 mL Bottle	P	Cool, 4°C	28 Days	
Sulfate	200 ^a	200 mL Bottle	P,G	Cool, 4°C	28 Days	
Sulfide	500	Quart Cube	P,G	See (e) below	7 Days	ORGANICS:
BOD	4000	Gallon Cube	P,G	Cool, 4°C	48 Hrs.	
COD	1000	Quart Cube	P,G	4°C, H ₂ SO ₄ pH<2	28 Days	6 mL
Oil and Grease	1000	Quart Jar	G	4°C, H ₂ SO ₄ pH<2	28 Days	6 mL
Phenolics	1000	Quart Jar	G	4°C, H ₂ SO ₄ pH<2	28 Days	6 mL
<u>ORGANICS:</u>						
Acid Herbicides	1000	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	14 days	
Insecticides	1000	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	7 days	
PCB's	1000	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	14 days	
Glyphosate	40	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	14 days	
Adipates/Phthalates	1000	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	14 days	
Endothall	40	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	14 days	
Carbamates	40	Liter Jar	AG	4°C, 80 mg/L Na ₂ SO ₃	28 days	Buffer pH<3
Diquat	1000	Liter Jar	AP	4°C, 100 mg/L Na ₂ SO ₃	7 days	
EDB/DBCP	40	VOC Vial	AG	4°C, 75 mg/L Na ₂ SO ₃	14 days	
PAH's	1000	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	7 days	
Trihalomethanes	40	VOC Vial	AG	4°C, 75 mg/L Na ₂ SO ₃	14 days	pH < 2 HCL
VOC's	40	VOC Vial	AG	4°C, Ascorbic acid	14 days	pH < 2 HCL
BTEX's	40	VOC Vial	AG	4°C, Ascorbic acid	14 days	pH < 2 HCL
Semi-volatiles	1000	Liter Jar	AG	4°C, 100 mg/L Na ₂ SO ₃	7 days	
TCLP	1000	Liter Jar	AG		14 days	

- (a) These analytes can all come from the same quart cubitainer
(b) These analytes can both come from the same preserved 500 ml HDPE bottle
(c) These analytes can both come from the same preserved 200 ml HDPE bottle
(d) These analytes can both come from the same 200 mL HDPE bottle
(e) Preserve this sample with 2 mL of zinc acetate plus NaOH to pH>9, cool 4°C
(f) These parameters can all optionally come from one 500 mL HDPE bottle preserved with 1:5 H₂SO₄ to pH<2 (2 mL)
(g) In all cases the volume of acid refers to 1:5 concentration if H₂SO₄ and concentrated if HNO₃
(h) P=plastic, G=glass, A=amber

If the sample is not accompanied with adequate identification, the sample may either be disposed of, held pending log in or may be returned to the sampler. This choice will be made at the laboratory's discretion. In any case, the sample will not be logged in until complete information is made available to the log in staff.

Conditions warranting disposal include but are not limited to:

- BOD samples collected between 1600 Friday and 1200 Monday.

- Samples which have greatly exceeded holding time (2 times or more of initial hold time period).

- Samples in improper containers (e.g., oil and grease in plastic).

- Samples improperly preserved (e.g., nitrate sample collected with nitric acid added).

This policy is implemented to maintain the integrity of the data produced by the Chemistry Division. Improper sample collection leads to inaccurate results. It is our intention to provide the data user with the most precise and accurate results possible given the current state of laboratory analytical capabilities.

SECTION VI
CALIBRATION PROCEDURES FOR CHEMISTRY AND
RADIOCHEMISTRY

Types of calibrations used for each method and the frequency are specified in the technical standard operating procedures. The source of standards and storage conditions are also described in the technical standard operating procedures.

SECTION VII ANALYTICAL PROCEDURES

EPA approved methodology is used for environmental analysis. Methods used for certified drinking water parameters are listed below. Methods listed at 40 CFR Part 136 are used for Clean Water Act monitoring and methods from SW-846 are used for evaluating solid and hazardous waste. AOAC and ASTM methods are used for regulatory analysis. Detailed analytical procedures are documented in the Analytical Standard Operating Procedures Manuals. These analytical procedures include the quality control checks that must be performed with each method.

Methods and Detection Limits for Certified Drinking Water Parameters

Inorganic Parameters

Parameter	Certified Method(s)	Detection Limit
Antimony	EPA 200.8	0.2 ug/L
Arsenic	EPA 200.8	0.2 ug/L
Barium	EPA 200.8	0.04 ug/L
Beryllium	EPA 200.8	0.2 ug/L
Cadmium	EPA 200.8	0.02 ug/L
Chromium	EPA 200.8	0.2 ug/L
Copper	EPA 200.8	0.04 ug/L
Cyanide	EPA 335.4	5 ug/L
Fluoride	Standard Methods 4500-F ⁻ C	10 ug/L
Lead	EPA 200.8	0.2 ug/L
Mercury	EPA 245.1	0.2 ug/L
Nickel	EPA 200.8	0.03 ug/L
Nitrate	EPA 353.2	5 ug/L
Nitrite	EPA 353.2	5 ug/L
Selenium	EPA 200.8	0.2 ug/L

Thallium	EPA 200.8	0.03 ug/L
Radium 226	EPA 600/4-75-008 (3/1976)	
Radium 228	EPA 600/4-80-032 (8/1980)	

Organic Parameters

Regulated Volatiles

Parameter	Certified Method	Detection Limit
Benzene	EPA 524.2	0.5 ug/L
Carbon Tetrachloride	EPA 524.2	0.5 ug/L
Chlorobenzene	EPA 524.2	0.5 ug/L
1,2-Dichlorobenzene	EPA 524.2	0.5 ug/L
1,4-Dichlorobenzene	EPA 524.2	0.5 ug/L
1,2-Dichloroethane	EPA 524.2	0.5 ug/L
1,1-Dichloroethylene	EPA 524.2	0.5 ug/L
cis-1,2-Dichloroethylene	EPA 524.2	0.5 ug/L
trans-1,2-Dichloroethylene	EPA 524.2	0.5 ug/L
Dichloromethane	EPA 524.2	0.5 ug/L
1,2-Dichloropropane	EPA 524.2	0.5 ug/L
Ethylbenzene	EPA 524.2	0.5 ug/L
Styrene	EPA 524.2	0.5 ug/L
Tetrachloroethylene	EPA 524.2	0.5 ug/L
Toluene	EPA 524.2	0.5 ug/L
1,2,4-Trichlorobenzene	EPA 524.2	0.5 ug/L
1,1,1-Trichloroethane	EPA 524.2	0.5 ug/L
1,1,2-Trichloroethane	EPA 524.2	0.5 ug/L

Trichloroethylene	EPA 524.2	0.5 ug/L
Vinyl Chloride	EPA 524.2	0.5 ug/L
Total Xylenes	EPA 524.2	0.5 ug/L
Total Trihalomethanes	EPA 524.2	

Regulated SOCs

Parameter	Certified Method (s)	Detection Limit
Ethylene Dibromide (EDB)	EPA 504.1	0.01 ug/L
1,2-Dibromo,3- (DBCP) Chloropropane	EPA 504.1	0.02 ug/L
2,4-D	EPA 515.1	0.1 ug/L
2,4,5-TP (Silvex)	EPA 515.1	0.2 ug/L
Dinoseb	EPA 515.1	0.2 ug/L
Pentachlorophenol	EPA 515.1	0.04 ug/L
Picloram	EPA 515.1	0.1 ug/L
Glyphosate	EPA 547	10 ug/L
Endothall	EPA 548.1	2.5 ug/L
Diquat	EPA 549.2	0.4 ug/L
Benzo (a) pyrene	EPA 550	0.02 ug/L
Dalapon	EPA 552.2, EPA 515.1	1.0 ug/L
Alachlor	EPA 525.2	
Atrazine	EPA 525.2	0.1 ug/L
Di(2-ethylhexyl)adipate	EPA 525.2	0.6 ug/L
Di(2-ethylhexyl)phthalate	EPA 525.2	0.6 ug/L
Endrin	EPA 525.2	0.01 ug/L

Endrin	EPA 508	0.01 ug/L
Heptachlor	EPA 525.2	0.04 ug/L
Heptachlor	EPA 508	0.04 ug/L
Heptachlor epoxide	EPA 525.2	0.02 ug/L
Heptachlor epoxide	EPA 508	0.02 ug/L
Hexachlorobenzene	EPA 525.2	0.1 ug/L
Hexachlorobenzene	EPA 508	0.1 ug/L
Hexachlorocyclopentadiene	EPA 525.2	0.2 ug/L
Hexachlorocyclopentadiene	EPA 508	0.2 ug/L
Lindane	EPA 525.2	0.02 ug/L
Lindane	EPA 508	0.02 ug/L
Methoxychlor	EPA 525.2	0.10 ug/L
Methoxychlor	EPA 508	0.10 ug/L
Simazine	EPA 525.2	0.07 ug/L
Chlordane	EPA 508.1	0.2 ug/L
Chlordane	EPA 508	0.2 ug/L
Toxaphene	EPA 508	1.0 ug/L
PCBs (screen)	EPA 508	0.05 ug/L
PCBs (total) as decachlorobiphenyl	EPA 508A	0.15 ug/L
Carbofuran	EPA 531.1	0.5 ug/L
Oxamyl	EPA 531.1	0.5 ug/L

Disinfection Byproducts

Parameter	Certified Method	Detection Limit
Bromoacetic acid	EPA 552.2	1 ug/L
Bromochloroacetic acid	EPA 552.2	1 ug/L
Chloroacetic acid	EPA 552.2	1 ug/L
Dibromoacetic acid	EPA 552.2	1 ug/L
Dichloroacetic acid	EPA 552.2	1 ug/L
Trichloroacetic acid	EPA 552.2	1 ug/L

Miscellaneous

Parameter	Certified Method	Detection Limit
Total Organic Carbon	Standard Method 5310C 19 th Edition Supplement	

SECTION VIII

DATA REDUCTION, VALIDATION, REPORTING, & VERIFICATION

Data reduction includes all processes which either change the form of expression or the quantity of data values. Conversion of raw data from laboratory tests to final results is accomplished in several ways. Some raw data is entered into the laboratory information management system (LIMS) which computes it into final results. Some raw data is converted to final results external to the LIMS. In these cases it is usually computed to final results either by means of linear regression in programmable calculators or in computer data systems specific to certain instrumentation. Formulas for calculating final results are included in each of the technical standard operating procedures. Software/computer calculation programs are validated by manual computation of results from the same raw data. Once this validation is performed, no errors should result unless incorrect data is entered.

When a result is entered into the LIMS either electronically or manually, it is compared to two parameters and a rounding function which ultimately determine how the data is stored in the system. The two parameters are *significant figures* and *decimal point offset*. Significant figures is fairly self-explanatory. It is recorded with the analyte definition in the form of an integer and this parameter is used to determine how many significant figures are stored in the result field. The default value is 3. Decimal point offset is an integer which describes the number of places past the decimal point which will be stored in the final result. The default is 2.

Rounding of digits is done according to the following rules:

When the first digit discarded is less than five, the last digit retained is not changed. When the first digit discarded is greater than five, or when the first digit discarded is a five and is followed by a digit other than zero, the last digit retained is increased by one. When the first digit discarded is exactly five followed only by zeros, the last digit retained is rounded upward if it is an odd number and is not adjusted if it is an even number.

Proper checks must be made at all data handling points between the analyst, who determines the raw data values, and the final report which summarizes the results. Raw data and results transcribed to the LIMS by analysts using work lists are verified by the analysts. A print out of the results entered/calculated is generated when the work list is distributed. The analyst is responsible for verifying this data/results entry and documents this on the print out sheet. Results entered into the LIMS without work lists and analyte result changes entered into the LIMS appear on the Data Change Log generated when the daily LIMS backup is done. The quality assurance coordinator contacts appropriate analysts to verify the results entered without work lists and the analyte result changes that appear on the Data Change Log.

Procedures and format for reporting results are outlined in the Log-In Procedures Manual.

SECTION IX TYPES OF QUALITY CONTROL CHECKS AND FREQUENCY OF THEIR USE

Quality control checks and their frequency are specified in the laboratory's written methods. These quality control checks include: initial demonstration of method capability and use of control charts; method detection limit calculations; calibration, internal and surrogate standards; instrument performance check standards; laboratory reagent blank, field reagent blank and trip blank; field and laboratory matrix replicates; laboratory fortified blank and laboratory fortified sample matrix replicates. Quality control and performance evaluation samples are analyzed periodically.

Qualitative identification/confirmation of contaminants is accomplished by various techniques. Most organic contaminants are confirmed by either use of dissimilar columns or mass spectrometry. Procedures for identification/confirmation are outlined in the laboratory's written analytical methods.

Participation in external quality control activities is essential to verify that the laboratory's internal quality control checks are effective and reliable. The laboratory participates in various external performance evaluation/check sample programs. These programs include:

Water Supply Studies

In order to maintain certification for drinking water parameters, the laboratory must report acceptable results, at least annually, for each certified parameter on water supply proficiency test studies purchased from an accredited private provider. Parameters included in these studies are at least those currently regulated under the Safe Drinking Water Act. A performance evaluation report is returned after the study is completed. The report indicates whether or not the results submitted by the laboratory are within the acceptance limits. The laboratory investigates any not-acceptable results and documents the findings of the investigation as well as implements any appropriate corrective action. The laboratory purchases appropriate portions of another water supply study if results for any parameters were not acceptable on the first study purchased during the calendar year.

EPA DMR-QA Studies

EPA conducts one Discharge Monitoring Report Quality Assurance Study each year. The DMR-QA studies are conducted to measure the proficiency of laboratories performing analyses under the National Pollutant Discharge Elimination System (NPDES). The North Dakota Department of Health (NDDOH) Chemistry Division is a contract laboratory for several permittees under the NPDES. The laboratory purchases the DMR-QA study samples from an accredited private provider, analyzes the samples, and reports results to each permittee for all analytes it analyzes for the permittee. The laboratory also submits results to the study provider as a water pollution study. Performance evaluation reports for each permittee are returned after the study is completed, and the laboratory receives a performance evaluation report for the corresponding water pollution study. The reports indicate whether or not the results submitted by the permittees are within the acceptance limits. The (NDDOH) Chemistry Division laboratory investigates any of its not-acceptable results and documents the findings of the investigation as well as implements any appropriate corrective action.

Radchem Studies

The laboratory participates in a drinking water radiochemistry proficiency test study for Radium 226 and Radium 228 at least annually from an accredited private provider. Acceptable performance for these analytes is required at least annually in order to maintain certification. A performance evaluation report is returned after the study is completed. The report indicates whether or not the results submitted by the laboratory are within the acceptance limits. The laboratory investigates any not-acceptable results and documents the findings of the investigation as well as implements any appropriate corrective action. If results for either parameter are not acceptable on the first study purchased during the calendar year the laboratory purchases a second study as a follow-up.

U.S. Geological Survey Standard Reference Sample Program

The laboratory participates in the USGS Standard Reference Sample Program twice each year. These samples cover inorganic parameters in the area of mineral, nutrient, and trace element chemistry. Out-of-limit results are an indication that analytical problems may be present so these are investigated and used to develop corrective action measures.

CDC Monthly Fluoridated Drinking Water Proficiency Testing

Monthly fluoridated drinking water proficiency testing samples at three concentration levels are issued by the national Center for Disease Control. Participation in this proficiency testing helps the laboratory maintain proficiency in the analysis of fluoride in drinking water. In addition, the laboratory is a referee laboratory for the program (one of eight in the nation). As a referee laboratory, the laboratory receives quarterly distribution of the samples prior to national distribution. The results of the laboratory's analysis of these quarterly samples are used to set the target values and target ranges for those samples that are distributed monthly.

American Association of Pesticide Control Officials Check Sample Program

Under this check sample program the laboratory receives six pesticide/herbicide check samples annually in a group. Samples usually consist of pesticide/herbicide formulations with some containing contaminants which need to be identified and quantitated. These check samples help the organic section evaluate and maintain its proficiency at analyzing pesticide/herbicide formulations and identifying and quantitating unknown contaminants. Statistical reports are returned to participating laboratories enabling them to determine if their results are within acceptable limits.

Magruder Fertilizer Check Sample Program

This check sample program sends check samples on a monthly basis. These check samples usually contain the primary plant food ingredients and occasionally secondary nutrients and micronutrients. An overall ranking of the laboratory's performance among all the participating laboratories is provided as well as a statistical report for each month's sample. These check samples help the laboratory maintain proficiency in analysis of commercial fertilizer products. Any out-of-acceptable-range results indicate that a problem exists that is then investigated and corrected.

Association of Florida Phosphate Chemists Fertilizer Check Sample Program

This check sample program sends check samples on a monthly basis. These check samples are used primarily to evaluate the laboratory's proficiency at analyzing nitrogen and phosphorus in fertilizer. A report is returned which shows statistics by the individual methods used and a compilation of each laboratory's results. If out-of-acceptable range results are reported, a potential problem exists that is then investigated and corrected.

American Association of Feed Control Officials Check Sample Program

This check sample program sends check samples on a monthly basis. These check samples are used primarily to evaluate the laboratory's proficiency at analyzing feed samples. The check samples usually contain all of the proximate components of feeds plus a number of other feed components such as minerals, vitamins, and medications. A report is returned which shows statistics by the individual methods used and a compilation of the results for each method. If out-of-acceptable-range results are reported, a potential problem exists that is then investigated and corrected.

Mid Continent Regional Group Gasoline Check Sample Program

The laboratory receives check samples once a month through this program. These check samples are used to evaluate the petroleum laboratory's proficiency at testing gasoline samples. Typical tests run on these samples are distillation, gravity, and octane rating. A statistical summary of the results from all participating laboratories is compiled and returned. If out-of-acceptable range results are reported, a potential problem exists that is then investigated and corrected.

Laboratory Proficiency Testing Program

The Laboratory Proficiency Testing Program (LPTP) is operated by Resource Technology Corporation at Laramie, Wyoming. This is a solid/hazardous waste performance evaluation program using natural matrix solids, sludges, and liquids to assess laboratory performance using SW846 methods. The laboratory signs up for any or all of the four organic/inorganic quarterly test samples depending on their applicability to the solid/hazardous waste testing normally done in the laboratory. At the completion of each quarterly test a report is sent to each participant. The reports contain a narrative summary, raw data summary, statistical analysis, lab Z-scores, and a graphical depiction. The laboratory's performance is rated against all other participating laboratories. This proficiency testing program enables the laboratory to assess and maintain its analytical proficiency with solid/hazardous waste methods.

SECTION X
SCHEDULES OF INTERNAL AND EXTERNAL SYSTEM AND
DATA QUALITY AUDITS AND
INTER LABORATORY COMPARISONS

EPA Region 8 performs a drinking water certification onsite evaluation of the Chemistry Division laboratory once every three years. This is essentially an external system and data quality audit. A summary report of the evaluation is sent to the laboratory. The laboratory responds to any deficiencies and recommendations indicated in the report.

Split samples are collected from certain permitted wastewater discharge facilities on a periodic basis and analyzed by the facility laboratory and the Chemistry Division laboratory.

Performance evaluation/check sample programs, which are essentially interlaboratory comparisons, are described in detail in section IX.

**SECTION XI
PREVENTIVE MAINTENANCE PROCEDURES, SCHEDULES,
AND AVAILABLE INSTRUMENTATION**

Preventive maintenance is an orderly program of positive actions (equipment cleaning, lubricating, reconditioning, adjusting, and testing) aimed at preventing failure of equipment or parts thereof during routine use. The main objective of the preventive maintenance program is to increase system reliability, thus decreasing downtime and increasing productivity.

A variety of some of the more inexpensive and more-likely-to-fail spare parts for most instrumentation are maintained in the laboratory.

The laboratory has the following major equipment and instrumentation available:

Inorganic Section

Instrument	Manufacturer	Model (number of units)
Alpha Beta Counter	Canberra	2404 (1)
Atomic Absorption (Flame)	Perkin Elmer	3100 (1)
Atomic Absorption (Furnace)	Perkin Elmer	Z5100 (1)
Automated Specific Ion Electrode System	Orion	940/960 (1)
Auto-Titrator	Mettler	DL53 (1)
Balance Top Loading	AND	HF 3000 G (1)
Balance Analytical	Cahn/Orion	CO-10 (1)
Balance Analytical	Mettler	AE160 (2)
Balance Top Loading	Ohaus	Precision Plus (1)
Block Digestor	Lachat	BD-46 (1)
Block Digestor	Environmental Express Hot Block	(1)
Centrifuge	International Equipment Co.	CL (1)

Centrifuge	Fisher	Marathon 22K (1)
Centrifuge	IEC	Model 7 (1)
Conductivity Meter	ATI/Orion	170 (1)
Desiccator	Hruden Lab Products	(1)
Distillation Unit	Lab Crest Midi Distillation Unit - Andrews Glass Co.	(1)
Distillation Unit	Labconco	RDU (1)
Drying Oven	Cervitor Kitchens	(1)
Drying Oven	Lab Line	L-C (1)
Fat Extractor	Tecator	1043 (1)
Flow Injection Analysis System & Ion Chromatograph	Lachat	Quickchem 8000 (1)
Flow Injection Analysis System	Lachat	Quickchem 8000 (1)
Freezer	Loudon	Upright (1)
Incubator	Precision Scientific	Thelco 32 MR (1)
Inductively Coupled Plasma Spectrometer	Perkin Elmer	Optima 3000 DV (1)
Inductively Coupled Plasma/Mass Spectrometer	Perkin Elmer/Sciex	Elan 5000 (1)
Ion Chromatograph System	Dionex	DX 500 (1)
Liquid Scintillation Counter	Packard	2500 TR/AB (1)
Microwave Digestion System	CEM	MDS2000 (1)
Microwave Digestion System	Milestone	Ethos Plus (1)
Muffle Furnace	Sybron/Thermolyne	(1)
Nitrogen Analyzer	Leco	FP-428 (1)
pH Meter	Fisher	Accumet 50 (1)

pH Meter	Fisher	AB15 (2)
pH Meter	Fisher	925 (1)
pH Meter	Orion	EA940 (1)
Refrigerator	Fisher	Isotemp (1)
Refrigerator	VWR Scientific	(1)
Refrigerator/Freezer	Kenmore	Kitchen Type (1)
Scintillation Counter	Randam	SC-5 (1)
Scintillation Counter	Ludlum	1000 (6)
Spectrophotometer	Sequoia Turner	390 (1)
Spectrophotometer UV visible	Beckman	DU-7 (1)
Total Organic Carbon Analyzer	Tekmar/Dohrman	Phoenix 8000 (1)
Turbidity Meter	Hach	2100N (1)

Organic Section

Instrument	Manufacturer	Model (number of units)
Accelerated Solvent Extractor	Dionex	ASE 200 (1)
Autosamplers (GC)	Hewlett Packard	7673 (8)
Autosampler (GC)	Varian	8085 (1)
Autosampler (LC)	Thermo Separation Products	AS 3000 (1)
Balance Top Loading	Mettler	PE11 (1)
Balance Top Loading	Ohaus	(1)
Chromatography Data System	Fisons	X-Chrom (1)

Detector (LC)	LDC	Spectro Monitor 5000 PDA (1)
Detector (LC)	Thermo Separation Products	FL 3000 Fluorescence (1)
Detector (LC)	Thermo Separation Products	FL 2000 Fluorescence (1)
Detector (LC)	ABI/Kratos	Spectroflow 757 uv (1)
Detectors (GC)	Hewlett Packard	ECD (7)
Detectors (GC)	Hewlett Packard	FID (3)
Detector (GC)	Hewlett Packard	PID (1)
Detector (GC)	Hewlett Packard	NPD (1)
Detector (GC)	OI	5320 ELCD (2)
Evaporator	Zymark	Turbo Vap II (3)
Evaporator	Zymark	Turbo Vap LV (1)
Food Chopper	Hobart	(1)
Freeze Dryer	Labconco	(1)
Gel Permeation Chromatography System	ABC	Autovap AS-2000 (1)
Gas Chromatograph	Hewlett Packard	5890 (7)
Gas Chromatograph/Mass Spectrometer System	Hewlett Packard	6890/5973N (1)
Gas Chromatograph/Mass Spectrometer	Thermo Quest Finnigan	Trace GC (1) Trace MS (1)
Gas Chromatograph/Mass Spectrometer System	Hewlett Packard	5890/5972 (1)
Gasoline Analyzer	Petro Spec	GS-100 (1)
Hydrogen Generator	Whatman	(2)
Liquid Chromatograph System	LDC Analytical	Consta Metric 4100 (1)

Liquid Chromatograph System	Varian	9010 (1)
Liquid Chromatograph Membrane Degasser	Thermo Separation Products	(3)
Liquid Chromatograph Post Column Reaction System	Pickering	PCX 5100 (1)
Nitrogen Generator	Peak	(1)
Purge and Trap System	Tekmar	3000 with AQUATek 50 Autosampler (1)
Refrigerator/Freezer	Fisher	Isotemp (2)
Refrigerator (Explosion Proof)	Lab Line	Frigid-Cab (1)
Refrigerator (Explosion Proof)	Fisher	Equa Therm (1)
Solid Matrix Extractor	Dionex	ASE 200 Accelerated Solvent Extractor (1)
Solid Phase Extraction System	Horizons	SPE DEX 4470 (1)
Solvent Controller	Dionex	ASE 200 Solvent Controller (1)
UV/Visible Spectrophotometer	Beckman	DU-7 (1)
Walk in Cooler	Norlake	(1)
Zero Air Generator	Peak Scientific	(1)

Service contracts are maintained on several of the laboratory's instruments and software systems and are summarized as follows:

Agilent Technologies

- Service, repair, and replacement of all malfunctioning parts for all HP gas chromatographs, detectors and autosamplers.

- Service, repair and replacement of malfunctioning parts for all gas chromatograph/mass selective detector systems plus 2 preventive maintenance visits per year. Also includes software revisions and upgrades and covers the printers, too.
- Software support - assist the end user.

Dionex

- Covers hardware and software for the ion chromatography system.

Lab Systems

- Covers all hardware and software of the Xchrom/Atlas chromatography data system including replacement, repair, and loaners.

Northwest Analytical

- LIMS software support.

Northern Balance Service

- Annual analytical balance service.

Packard Instrument

- Covers repair and parts replacement for liquid scintillation counter plus one preventive maintenance call per year.

Perkin Elmer

- Covers service, repair, and replacement of malfunctioning parts for all atomic absorption units, the inductively coupled plasma emission spectrometer (ICP) and the ICP/MS. This includes the autosamplers and other peripherals.

Lachat

- Covers all repairs and loaner service for the QuikChem 8000 systems including the pump, autosampler and chemistry modules.

OI Analytical

- Covers repairs and parts for two ELCD detectors.

Thermo Finnigan

- Covers parts and labor necessary to maintain the Trace GC/MS system in good working order. Preventive maintenance visits are covered in the plan in accordance with Thermo Finnigan guidelines.

SECTION XII CORRECTIVE ACTION CONTINGENCIES

When unacceptable results are obtained on performance evaluation samples, analysts review the records for any errors that might explain the not-acceptable results. The record reviews are documented on a response form which includes space for listing suggested corrective action. The response forms are included with the performance evaluation study records retained by the quality assurance coordinator. Follow-up activities might include analysis of a quality control sample to verify that the corrective action was successful.

Analysts are responsible for evaluating the quality control checks they run with each method. If the results of the quality control checks are not-acceptable, the analyst must take appropriate corrective action. First, the analyst should rerun the originally prepared quality control sample, if possible to determine if the original analysis is the source of the out-of-control situation. If the rerun test result indicates that the system is back in control, this fact should be documented on the control chart and/or work list. If the out-of-control situation still exists after the rerun, the quality control sample should be reprepared to determine if the problem is with preparation. If reparation of the quality control sample brings the system back into control, this fact should be documented on the control chart and/or work list. If the system is still out of control, the next step is to reprepare the standards and the standard curve. If the curve is significantly different from the previous curve, the samples and quality control checks should be rerun. If the quality control checks show that the system is back in control, this should be documented on the control chart and/or work list. If the system is still out of control, the supervisor, division director, or the quality assurance coordinator should be contacted.

If a laboratory employee discovers an error in any results that have been entered into the laboratory information management system, a correction is made and a new report is generated. If an outsider claims there is an error in a result or results on an analytical report, the claim is investigated and if an error is confirmed, the error is corrected and a new report is generated.

SECTION XIII RECORD KEEPING PROCEDURES

Records of laboratory analyses are retained in notebooks, on worksheets, and on hard copy print outs from instrument or auxiliary data systems. Electronic files from some instruments are retained in the instruments and electronic files from auxiliary data systems are archived on system back-up tapes and a record is retained of archived files. Hard copies and other paper records are stored in each laboratory, either in three-ring binders or in boxes, for a period of approximately one year and then the records are sent to long-term storage off site. Some analytical areas may have variations of the above record keeping procedures.

Changes in data on the LIMS system are followed through audit trails beyond the first level of security. The log in personnel generate data change logs at the end of each day. These change logs list any changes in results done without work lists and results entered without work lists. LIMS data change logs are retained by the quality assurance coordinator.