

Regulation 85 Nutrient Sampling and Analysis Plan

AF CURE

Fountain Creek and Arkansas River

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Regulation 85 Sampling and Analysis Plan

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List of Abbreviations

AWQMS	Ambient Water Quality Monitoring System
CDPS	Colorado Discharge Permit System
CDSN	Colorado Data Sharing Network
CDWR	Colorado Division of Water Resources
Commission	Colorado Department of Public Health and Environment, Water Quality Control Commission
Division	Colorado Department of Public Health and Environment, Water Quality Control Division
EPA	Environmental Protection Agency
GPS	Global Positioning System
Major Discharge	>1 MGD domestic wastewater treatment works
MDL	Method Detection Limit
MGD	Million Gallons per Day
Minor Discharge	≤1 MGD domestic wastewater treatment works
PQL	Practical Quantification Limit
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RL	Reporting Limit
SAP	Sampling and Analysis Plan
TIN	Total Inorganic Nitrogen
TKN	Total Kjeldahl Nitrogen
TN	Total Nitrogen
TP	Total Phosphorus
USGS	United States Geological Survey

Section 1

SAP OVERVIEW

1.1 Background

This Sampling and Analysis Plan (SAP) was developed to meet the requirements of Colorado Department of Public Health and Environment Water Quality Control Commission (Commission) Regulation 85, Nutrients Management Control Regulation 5 CCR 1002-85. The monitoring, as required in Regulation 85, is to evaluate the effectiveness of the control regulation and to determine the sources and load of nutrients at selected locations, and eventual implementation of appropriate and necessary source controls. The Colorado Department of Public Health and Environment, Water Quality Control Division (Division) will be analyzing all monitoring information to determine the sources and load of nutrients at selected locations. As the sampling effort required under Regulation 85 is mainly intended to characterize point source dischargers, there will be gaps in the data with regard to characterization of other sources of nutrients. Additional data, outside of the scope of this SAP, would be required to characterize watershed nutrient loading or for use in site-specific standards.

1.2 Description

Regulation 85 requires domestic and selected non-domestic wastewater treatment facilities to develop, implement, and document a routine water monitoring program. The monitoring program is designed to characterize the load (coincident flow and concentration) of nutrients in the discharge, the concentrations in the receiving water above the discharge, and the load of nutrients in the river or stream below the discharge.

1.3 Time Line

The certification, see Appendix A, was submitted to the Division and data collection began no later than March 1, 2013. For cooling towers, data collection began in November 2012 will continue no longer than two years after the first sample was collected (through October 2014).

1.4 Monitoring Requirements

1.4.1 Effluent Monitoring

1.4.1.1 Locations

Sampling for nutrients is required in the effluent before it is discharged into the receiving water body at the location where monitoring is performed to satisfy other Colorado Discharge Permit System (CDPS) permit requirements.

1.4.1.2 Parameters

At minimum, sufficient data are collected to calculate:

- Total Nitrogen (TN) - Test for Total Kjeldahl Nitrogen (TKN) and Nitrate+Nitrite Nitrogen
- Total Inorganic Nitrogen (TIN) - Test for Ammonia and Nitrate+Nitrite Nitrogen
- Total Phosphorus (TP)
- Flow - The total daily effluent discharge flows (in gallons or million gallons per day) are collected at the same time as the nutrient concentrations are measured.

1.4.1.3 Frequency

- Samples are collected a minimum of six times a year (every two months).
- Samples are collected a minimum of monthly.

1.4.1.4 Sample Types

Effluent samples types are identified in Table 4-1 of Section 4.

1.4.2 Stream Monitoring

1.4.2.1 Locations

Sampling for nutrients is required in the receiving water body at the following locations:

- Upstream of the discharge.
- At the closest active Colorado Division of Water Resources (CDWR) or United State Geological Survey (USGS) gaging station with daily flow available throughout the year downstream of the discharge's mixing zone.

1.4.2.2 Parameters

At a minimum, samples are analyzed for:

- Total Nitrogen (TN) - Test for Total Kjeldahl Nitrogen (TKN) and Nitrate+Nitrite Nitrogen
- Total Phosphorus (TP)
- Flow – Daily average streamflow (cfs) on the day instream samples are collected is obtained where an established gaging station with a dataloger is present.

1.4.2.3 Frequency

Samples are collected a minimum of monthly.

1.4.2.4 Sample Types

Ambient stream samples are collected as grab samples.

Section 2

Analytical Methods

2.1 Analytical Method Detection Limits

The analytical methods for ambient monitoring must have method detection limits (MDL) no greater than the limits listed below:

Total Phosphorus	0.01 mg/L
Nitrate + Nitrite Nitrogen	0.02 mg N /L
Nitrate Nitrogen	0.02 mg N/L
Nitrite Nitrogen	0.02 mg N/L
Total Kjeldahl Nitrogen	0.1 mg N /L
Total Nitrogen	0.1 mg/L

All results at or above the MDL are reported for ambient data. Data measured between the MDL and the lab practical quantitation limit (PQL) are reported and flagged with “J” as qualified data.

The analytical method for all effluent monitoring must have a PQL no greater than the limits listed below:

Total Phosphorus	0.01 mg/L
Nitrate + Nitrite Nitrogen	0.5 mg N /L
Nitrate Nitrogen	0.05 mg N/L
Nitrite Nitrogen	0.01 mg N/L
Total Kjeldahl Nitrogen	0.5 mg N /L
Total Nitrogen	0.5 mg/L
Ammonia Nitrogen	0.05 mg N/L

A numerical value must be reported for all data equal to or greater than the PQL. Data less than the PQL are reported as less than the PQL.

2.2 Analytical Methods

All samples are collected using standard sampling procedures, sample bottles, and preservatives as prescribed by the method. Samples are delivered to the lab and are analyzed within analysis holding times as specified in Table 1A in 40 CFR Part 136 (see Appendix B). All methods used for analysis of samples are listed in the most current version of 40 CFR Part 136 or are approved by the Division (see Appendix B). The lab follows and meets method and general lab quality control procedures including the use of method blanks, sample spikes, known value standards, and duplicate sample analysis.



Section 3

Data Reporting

3.1 Reporting Procedures

All data collected are maintained in an electronic form and will be submitted to the Division via the Data Sharing Network by April 15, 2014 and then each April 15th thereafter. AF CURE will notify the Division by April 15th of each year that the data are available in the DSN and all relevant data will be accessible to the public. The submission will include geographic locations of sampling points, CDPS permit number, method of sample collection, method detection limit, practical quantitation limit, sample results, sample units, and name and identification of the stream flow gage used in ambient flow calculation.

When calculated flow data are reported, the fact that the results are calculated will be noted in the notes column. Alternatively, flows can be entered for measured locations and flow calculations can be left for the Division to complete, where needed.

Section 4

Sampling Protocol

4.1 Sampling Locations

The sampling locations below were selected to comply with the requirements of Regulation 85. Figure 4-1 is a map of the sampling locations for AF CURE. Descriptions and GPS coordinates for each sampling location are provided in Table 4-1 below.

Table 4-1. AF CURE Sampling Locations, Flow Monitoring Methods, and Sampling Frequency					
Facility/Sampling Location	Location ID	Latitude	Longitude	Flow Monitoring Method	Sampling Frequency
Tri-Lakes WWTF					
Monument Creek at Arnold Ave. Bridge	Arnold Ave.	39° 5' 15"	-104° 52' 45"	USGS gage 07103755 (Monument Creek below Monument Lake)	Monthly
Discharge from Tri-Lakes WWTF	Tri-Lakes WWTF EFF	39° 4' 12.18"	-104° 52' 28.32"	Tri-Lakes WWTF Effluent Flow	Monthly
Monument Creek at Baptist Rd. Bridge	Baptist Rd.	39° 3' 22.14"	-104° 51' 46.2"	USGS gage 07103780 (Monument Creek above N. Gate Blvd. at USAFA) minus discharge flow from Upper Monument WWTF	Monthly
Upper Monument Creek Regional WWTF					
Monument upstream of Upper Monument WWTF	Upstream UMCR	39° 2' 31.8402"	-104° 51' 9.0684"	USGS gage 07103780 (Monument Creek above N. Gate Blvd. at USAFA) minus discharge flow from Upper Monument WWTF	Monthly
Upper Monument WWTF	UMCR Effluent	39° 2' 34.5582"	-104° 51' 9.7194"	Upper Monument WWTF Effluent Flow	Monthly

Mon.Creek at USGS Station 07103780	Downstream UMCR	39° 1' 51.7"	-104° 50' 47.8"	USGS gage 07103780 (Monument Creek above N. Gate Blvd. at USAFA)	Monthly
JD Phillips WWTP					
Monument Creek upstream of JD Phillips WWTP discharge	USGS gage 07104000	38° 53' 38"	-104° 49' 20"	USGS gage 07104000	Monthly
JD Phillips WWTP contribution to Monument Creek.		38° 53' 24"	-104° 49' 30"	JD Phillips Effluent Flow	Monthly
Monument Creek downstream of JD Phillips WWTP discharge	USGS gage 07104905	38° 53' 24"	-104° 49' 26"	USGS gage 07104905	Monthly
Las Vegas WWTP					
Fountain Creek upstream of Las Vegas WWTP discharge	USGS gage 07105500	38° 48' 59"	-104° 49' 20"	USGS gage 07105500	Monthly
Las Vegas WWTP contribution to Fountain Creek.		38° 48' 44.0202"	-104° 48' 37.3716"	Las Vegas WWTP Effluent Flow	Monthly
Fountain CR BLW Janitell Rd. BLW Colo Springs, CO	USGS gage 07105530	38° 48' 11"	-104° 47' 43"	USGS gage 07105530	Monthly
Fountain Sanitation WWTP					
Fountain Creek upstream of Fountain Sanitation WWTP discharge	FSD US	38° 39' 51.6"	-104° 41' 44.2"	Fountain Creek Transit Loss Model Mode 22	Monthly
Fountain Sanitation WWTP contribution to Fountain Creek.		38° 39' 42.8"	-104° 41' 56.1"	Fountain Sanitation WWTP Effluent Flow	Monthly
Fountain Creek downstream of Fountain Sanitation WWTP discharge	FSD DS – Owner & Hall Ditch Diversion	38° 38' 51.9"	-104° 41' 29.6"	Fountain Transit Loss Model Mode 23	Monthly

Security Sanitation District WWTF					
Fountain CK at Colo Springs	SSD Upstream	38° 44' 48"	-104° 44' 40"	USGS gage 07105800 (Fountain Creek at Security, CO; Carson Road Bridge) minus discharge flow from SSD WWTF	Monthly
SSD WWTF contribution to Fountain Creek.	SSD Effluent	38° 44' 6.9"	-104° 44' 14.76"	SSD WWTF Effluent Flow	Monthly
Fountain Creek @ SSD WWTF	SSD Downstream	38° 43' 46"	-104° 44' 00"	USGS gage 07105800 (Fountain Creek at Security, CO; Carson Road Bridge)	Monthly
Widefield Water and Sanitation District					
Fountain Creek upstream of Widefield WSD discharge	WWSD U	38° 43' 4.95"	-104° 43' 31.57"	Sum flows at: USGS 07105800 (Fountain Cr. Near Security), USGS 07105820 (Clover Ditch below Ft. Carson WWTP) OR Ft. Carson WWTP flows if the USGS in not operational as the Ft. Carson WWTP comprises most of the flow in Clover Ditch, and DWR Cruse Gulch through Willow Springs Ponds.	Monthly
Widefield WSD contribution to Fountain Creek.	WWSD E	38° 43' 7.68"	-104° 43' 29.75"	Widefield WSD Effluent Flow	Monthly
Fountain Creek downstream of Widefield WSD discharge	WWSD D	38° 42' 50.55"	-104° 43' 21.60"	Calculated upstream flow + Widefield WSD effluent flow.	Monthly
Lower Fountain MSDD					
Un-named #2 upstream of LMFSDD discharge	LFMSDD US	38° 37' 28.5"	-104° 39' 25.8"	Flow measurement at road culvert	Monthly
LFMSDD contribution to Un-named #2.	LFMSDD Discharge	38° 37' 7.4"	-104° 39' 46.8"	LFMSDD Effluent Flow	Monthly

Un-named #2 downstream of LMFSDS discharge	LFMSDD DS	38° 36' 52.7"	-104° 39' 45.7"	LFMSDD US and LFMSDD Discharge	Monthly
Woodman Hills WWTP					
Drop structure upstream of Woodman Hills WWTP Discharge	Drop structure upstream of Woodman Hills WWTP Discharge	38° 58' 11.96"	-104° 35' 30.72"	Calculated	Monthly
Woodman Hills WWTP 001A Discharge	001A	38° 58' 11.65"	-104° 35' 30.56"	Woodman Hills WWTP Effluent Flow	Monthly
Receiving stream crosses Garrett Road	Receiving stream crosses Garrett Road	38° 54' 40.09"	-104° 34' 58.71"	Calculated	Monthly
Pueblo West Metro District WWTP					
Wildhorse Creek above Pesthouse	Wildhorse Creek above Pesthouse	38° 17' 46.80"	-104° 38' 55.78"	3-inch flume manually read.	Monthly
No Name above Pesthouse	No Name above Pesthouse	38° 18' 34.84"	-104° 40' 19.88"	3-inch flume manually read.	Monthly
Discharge from PWMD WWTP	Effluent from WWTP	38° 18' 51.19"	-104° 40' 29.69"	PWMD WWTP Effluent Flow	Monthly
Arkansas River at Moffat Street	25RWMOFFAT	38° 15' 13"	-104° 36' 20"	USGS gage 07099970 (Arkansas River at Moffat Street)	Monthly
Pueblo WRF					
Arkansas River sampling above Pueblo WRF is comprised of three sample locations:				Sum flows at: USGS 07099970 (Arkansas River at Moffat Street), USGS 07099973 (Runyon Lake Outlet), and DWR FOUOUCO (Fountain Creek Above Confluence)	
Arkansas at Moffat	25RWMOFFAT	38° 15' 13"	-104° 36' 20"		Monthly
Runyon Lake Outlet	23RWRUNFLout	38° 15' 14"	-104° 35' 45"		Monthly
Fountain Creek above Confluence	28RWFTNCO	38° 15' 19.2"	-104° 35' 26.1"		Monthly

Discharge from Pueblo WRF	70EFFsampler	38° 15' 21.44"	-104° 34' 33.9"	Pueblo WRF Effluent Flow	Monthly
Arkansas River at CS-10	23RWCS10	38° 15' 35.73"	-104° 33' 28.4"	Calculated upstream flows + Pueblo WRF effluent flow	Monthly

4.2 Sampling Parameters

The sampling parameters are provided in Table 4-1. When a calculation method is used to determine the TN or TIN, the fractions as well as the calculated total will be reported.

Table 4-2. Sampling Parameters											
Facility	Effluent Sampling Method	Analytical Laboratory	Laboratory Method								
			Total Phosphorus	Total Nitrogen	Nitrate Nitrogen	Nitrite Nitrogen	Nitrate+Nitrite Nitrogen	Ammonia Nitrogen	Total Kjeldahl Nitrogen	Total Inorganic Nitrogen	
Tri-Lakes WWTF	Composite	Tri-Lakes WWTF		TKN+NO ₃ +NO ₂	SM 4500-NO ₂ B			SM 4500-NO ₃ E	SM 4500-NH ₃ D		NH ₃ +NO ₂ +NO ₃
		SGS Mineral Services (Denver)	SM 4500-P E							SM 4500-Norg B	
Upper Monument Creek Regional WWTF	Composite	ACZ Laboratories	SM 365.1							SM 351.2	
		UMWWTF		TKN+NO ₃ +NO ₂				SM4500-NO ₃	SM4500-NH ₃		
JD Phillips WWTP	Composite	USGS for ambient. Colorado Springs Utilities lab for effluent	Colorado Springs Utilities lab: 200.7	Colorado Springs Utilities: Lachat 10-107-04-4							Colorado Springs Utilities: Lachat 10-107-04-4
Las Vegas WWTP	Composite	USGS for ambient. Colorado Springs Utilities lab for effluent	Colorado Springs Utilities lab: 200.7	Colorado Springs Utilities: Lachat 10-107-04-4							Colorado Springs Utilities: Lachat 10-107-04-4

Fountain Sanitation WWTP	Composite	Colorado Analytical Laboratories	EPA 365.1	Calculation	EPA 300.0	EPA 300.0	Calculation	SM 4500-NH3-G	SM 4500-Norg-B	Calculation
Security Sanitation District WWTF	Composite	Accutest	SM4500-P, B/E-2011	SM4500N-2011	EPA 300.0, RV 2.1	EPA 300.0, RV 2.1	EPA 300.0, RV 2.1	SM4500N H3 D-2011	EPA 351.2	SM4500N-2011
Widefield Water and Sanitation District	Composite	SGS / In-house	SM 4500 - PE	TKN+NO3 +NO2	4110 B	4110 B	4110 B	SM 4500 NH3	SM 4500 - Norg	
Lower Fountain MSDD	Composite	Colorado Analytical Laboratories	EPA 365.1	Calculation	EPA 300.0	EPA 300.0	Calculation	SM 4500-NH3-G	SM 4500-Norg-B	Calculation
Woodman Hills WWTF	Composite	Colorado Analytical	EPA 365.1	TKN+NO3 +NO2	EPA 300.0	EPA 300.0	Calculation	SM 4500-NH3-G	SM 4500-Norg-B	
Pueblo West Metro District WWTP	Composite	ACZ Laboratories	M365.1 - Auto Ascorbic Acid (digest)	TKN+NO3 +NO2			EPA 353.2	EPA 350.1	EPA 351.2	NH3+NO2 +NO3
Pueblo WRF	Composite	Pueblo WRF (1)	EPA 365.1	Lachat 10-107-04-4			EPA 353.2	4500-NH3D-1997		

(1) Pueblo may send February and March 2013 samples to Test America to allow time for the Lachat TN Method to be set up and verified prior to use. If so, the TN would be a calculation of TKN (EPA 351.2) and NO3+NO2 (EPA 353.2).

4.3 Sampling Techniques

The sampling is conducted by AF CURE members using the following protocols. Samples are collected in containers provided by the laboratories and delivered for analysis per laboratory recommendations.

Careful precision in the laboratory cannot compensate for improperly collected samples. Inadequate field techniques, poor equipment, and improper sample preservation will have a greater effect on the testing outcome than errors from variation in laboratory analyses.

4.3.1 Field Notes, Sample Labeling, and Chain of Custody

Field notes are taken for all sample sites and recorded in a bound field notebook. Information recorded includes: identification of the monitoring site; date and time of sampling, identity of the sampler(s); description of the type of samples taken; method of sampling; results

of any field analyses; description of the weather, including percent cloud cover and air temperature; description of the site appearance; and any unusual conditions observed.

Collected samples are designated by sample location using the location ID identified above. Each sample container is individually labeled, with the label affixed directly to the bottle or bag itself with the preservative and analysis to be performed printed on the label. Additional sampling information, date, time, location, and sampler initials, is also written on the label with indelible waterproof ink.

Chain of custody (COC) documentation identifies sample containers and provides a complete inventory of all containers in a sample set, and will provide an audit trail identifying the persons who have custody of a sample in order and the exact date and time when custody was relinquished from one person to the next. A COC will be filled out for each sampling event (See Appendix D for sample).

4.3.2 Sampling Containers

The lab provides the containers and any shipping materials necessary to maintain sample integrity from the time of collection through analysis. Table II of 40 CFR section 136.3 defines specific materials, preservation techniques, and holding times (see Appendix B)

4.3.3 General Sampling Recommendations

Following are general recommendations for water quality sampling.

- Since sampling is taking place year round, it is possible that instream sampling may not be safe at certain times. If alternative methods of sample collection are not possible, it is up to the discretion of the individual sampling personnel whether sampling will take place during high flows. The turbidity of the stream, visual flow, and previous rain events may also affect sampling schedule.
- When wading, collect samples upstream from the body and avoid disturbing sediments in the immediate area of sample collection.
- Sampling at or near structures (e.g., dams, weirs, or bridges) may not provide representative data because of unnatural flow patterns. If you have to collect from a bridge, sample on the upstream side in the center of the main flow.
- Collect grab samples within the top 12 inches of the water column, but avoid skimming the surface of the water during collection.
- Where practical, use the actual sample container as the collection device (direct grab). If a direct grab sample cannot be collected, ensure that the intermediate sample container is well rinsed with site water before sample collection.

4.3.3.1 Stream Grab Sample Technique

Grab samples will be collected for analysis of ambient water quality. Grab sample technique is summarized as follows:

- Use an unpreserved sample container to collect the sample. Rinse with sample site water.
- If using prepreserved sample bottles, collect sample water (in same manner described below) in a clean carboy/sample collection container that is rinsed with sample site water. Fill the bottles from that carboy.
- Take sample from area representative of the flow conditions at that site.
- Remove the container cap and slowly submerge the container, opening first, into the water.
- Invert the bottle so the opening is facing toward the water and parallel to water flow. Allow water to run slowly into the container until filled.
- Return the filled container quickly to the surface.

- Pour out a small volume of sample away from and downstream of the sampling location. This procedure allows for addition of preservatives (if using) and sample expansion.
- Add preservatives (provided by the analytical laboratory), if required, securely cap container, label, and complete field notes and COC.
- If preservatives have been added, invert the container several times to ensure sufficient mixing of sample and preservatives.

4.3.3.2 Effluent Composite Sample Technique

Composite samples will be collected for effluent samples.

Use an aliquot of well mixed effluent composite and place in appropriate container.

- Add preservatives (provided by the analytical laboratory), if required, securely cap container, label, and complete field notes and COC.
- If preservatives have been added, invert the container several times to ensure sufficient mixing of sample and preservatives.

4.3.3.3 Flow Determination

Flow is an important tool in assessing water quality. Measurements will be recorded to help understand flow regimes within the watershed and to determine constituent loading. Daily average flow in cubic feet per second (cfs) from either a United States Geological Survey (USGS) or Colorado Division of Water Resources (CDWR) gage station(s) will be determined on the day samples are collected. Prior to each field visit, the USGS/CDWR website will be checked to confirm that the gage is active and flow data are available. Flow will be reported based the stream gage flow or will be calculated based on the methods provided in Section 4.1.

Total outfall flow will be reported in gallons or million gallons per day (gpd or MGD) on the day the sample is collected.

Where USGS or DWR flow data are not available, alternative flow calculations have been presented to the Division for approval.

4.4 QA/QC

All information produced must be of reliable and documented quality. The primary means for ensuring data quality is through implementation of QAPPs. These QA/QC steps are recommendations. Actual QA/QC steps completed by each data collection entity, if different from the steps below, are documented in each entity's SOPs or sampling plans specific to their facility.

Field QA/QC includes thorough cleaning of sampling equipment, use of appropriate sample containers, and maintaining COC procedures. Analytical QA/QC measures are also followed by the laboratories and include equipment blanks and spikes. Analytical QA/QC results are provided by the laboratory.

Field duplicates and field blanks should be collected, one for every 20 samples collected. The field duplicate is collected immediately after the primary sample is collected at the site. Field blanks are taken by pouring reagent water from the laboratory into sample bottles at the site. Reagent water would need to be requested from the laboratory. Field duplicates/blanks are labeled as separate samples to avoid confusion and to provide an unbiased blind evaluation.

Laboratory QC meets the requirements specified by each analytical method.

4.5 Data Review

Data review consists of reviewing the data package received from the contracted laboratories to ensure the package is complete and consistent. Actual Data Review steps completed by each data collection entity, if different from the steps below, are documented in each entity's SOPs or sampling plans specific to their facility. The following data review procedures are performed following receipt of each data package.

Step 1

1. Review the data set for completeness. Confirm that all sample sites and constituents are reported or that there is an explanation for each missing data point.
2. Review the data report. Confirm that all titles, labels, column headings, and footnotes are accurate and complete. Confirm that all constituents are reported in proper units.
3. Review the date and time documentation. Confirm that the sample dates and times are consistent with the date and time received in the laboratory. Confirm that the dates and time for analysis are consistent with the dates and times of the analysis. Confirm that the holding times were not violated, based on a comparison of sampling and analysis date and times.

Step 2

1. Review all values that are reported as "None Detected." Confirm that the analytical detection limits are low enough to accomplish project goals. Confirm that all values are either reported as values or less than the detection limit. Confirm that the detection limit is used consistently on all samples.

Step 3

1. Review data for internal consistency. Confirm that values have a logical relationship to one another. Confirm that values are within the historical range of data for a given site and constituent. Confirm that values vary logically according to known conditions, such as seasonal temperature and presence or absence of dilution flows.
2. Review the internal and external quality control results. Confirm that spike recovery percentages on matrix spikes, relative percent difference on laboratory duplicates, and percent error on known laboratory standards were within acceptance limits. Confirm that digestion blanks, reagent blanks, and method blanks do not contain concentrations of analyte that interfere with interpretation of data.

Appendix A: SAP Certification

Certification of Sampling and Analysis Plan for Nutrients

Certification of Sampling and Analysis Plan for Nutrients As Required by Regulation 85

Facility Name: _____

Permittee: Organization Formal Name: _____

Permit Number(s): _____

Permitted Discharge Flow: _____

This certifies that the facility named above has current Sampling and Analysis Plan ("SAP") in place for the monitoring of nutrients as required by Regulation #85. The SAP includes, but is not limited to: plant effluent monitoring location (latitude/longitude) where monitoring is performed; effluent parameters sampled (at a minimum, sufficient data, including flows, shall be collected to calculate TN, TIN, and TP load); daily average effluent discharge; date and time of sample collection; stream nutrient monitoring locations (latitude/longitude) upstream and downstream of the discharge; latitude/longitude coordinates for the closest active Colorado Division of Water Resources or United States Geological Survey (USGS) gaging station with daily flow available throughout the year downstream of the discharge's mixing zone or in lieu of the closest downstream Division of Water Resources or USGS gaging station, facilities may take part in collaborative watershed-based monitoring; parameters sampled (at a minimum, sufficient data shall be collected to calculate TN and TP load); and date and time of sample collection.

This SAP is available upon request by the Water Quality Control Division or other interested parties.

Signature of Legally Responsible Party_____
Date_____
Name (Printed) and Title

Additional Information Requested on the Back.

Additional Information Requested Regarding Sampling Locations

Location of Discharge:

Waterbody _____

Latitude/Longitude _____

Upstream monitoring location (if applicable):

Waterbody/Station Number _____

Latitude/Longitude _____

Downstream monitoring location (if applicable):

Waterbody/Station Number _____

Latitude/Longitude _____

Collection Agency of downstream gaging station (if applicable): _____

The SAP to meet the Regulation # 85 monitoring requirement for this facility is part of a collaborative watershed-based monitoring program.

Group/Watershed Name: _____ AF CURE _____

Contact Person for SAP (if needed): _____ Sarah Reeves _____

Contact Telephone # and/or e-mail address: _____ (303) 239-5411 sreeves@brwncald.com _____

Appendix B: 40 CFR Part 136 Excerpt

Analytical Methods, Sample Holding Times, Sample Bottles, and Preservatives

Parameter/Preservative/ Holding Time/Bottle Type	Methodology	EPA	Standard methods	ASTM	USGS/AOAC/ Other
4. Ammonia (as N), mg/L	Manual distillation or gas diffusion (pH > 11), followed by any of the following:	350.1, Rev. 2.0 (1993)	4500-NH ₃ B-1997		973.49 ³ .
Add H ₂ SO ₄ to pH < 2, store at 4°C for up to 28 days	Nesslerization			D1426-08 (A)	973.49 ³ , I-3520-85. ²
	Titration		4500-NH ₃ C-1997		
500-mL; plastic or glass	Electrode		4500-NH ₃ D-1997 or E-1997	D1426-08 (B)	
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods		4500-NH ₃ F-1997		See footnote. ⁶⁰
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods	350.1 ³⁰ , Rev. 2.0 (1993)	4500-NH ₃ G-1997		I-4523-85. ²
			4500-NH ₃ H-1997.		
	Automated electrode	Ion Chromatography		D6919-09	See footnote. ⁷
31. Kjeldahl Nitrogen ⁵ -Total, (as N), mg/L	Manual digestion ²⁰ and distillation or gas diffusion, followed by any of the following:		4500-N _{org} B-1997 or C-1997 and 4500-NH ₃ B-1997	D3590-02(06) (A)	
Add 2mL/L H ₂ SO ₄ , analyse ASAP or store at 4°C for up to 28 days	Titration		4500-NH ₃ C-1997		973.48. ³
	Nesslerization			D1426-08 (A)	
500-mL; plastic or glass	Electrode		4500-NH ₃ D-1997 or E-1997	D1426-08 (B)	
	Semi-automated phenate	350.1 Rev 2.0 1993	4500-NH ₃ G-1997.		
			4500-NH ₃ H-1997		
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods		4500-NH ₃ F-1997		See footnote. ⁶⁰

Parameter/Preservative/ Holding Time/Bottle Type	Methodology	EPA	Standard methods	ASTM	USGS/AOAC/ Other
	Automated Methods for TKN that do not require manual distillation				
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation)	351.1 (Rev. 1978) ¹			I-4551-78. ⁸
	Semi-automated block digester colorimetric (distillation not required)	351.2, Rev. 2.0 (1993)	4500-N _{org} D-1997	D3590-02(06) (B)	I-4515-91. ⁴⁵
	Block digester, followed by Auto distillation and Titration				See footnote. ³⁹
	Block digester, followed by Auto distillation and Nesslerization				See footnote. ⁴⁰
	Block Digester, followed by Flow injection gas diffusion (distillation not required)				See footnote. ⁴¹
38. Nitrate (as N), mg/L Store at 4°C for up to 48 hours	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997)	4110 B-2000 or C-2000	D4327-03	993.30. ³
100-mL; plastic or glass	CIE/UV		4140 B-1997	D6508-00(05)	D6508, Rev. 2. ⁵⁴
	Ion Selective Electrode		4500-NO ₃ ⁻ D-2000		
	Colorimetric (Brucine sulfate)	352.1 (Issued 1971) ¹			973.50, ³ 419D ^{1,7} , p. 28. ⁹
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40)				See footnote. ⁶²
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction, Manual		4500-NO ₃ ⁻ E-2000	D3867-04 (B)	
Add H ₂ SO ₄ to pH < 2, store at 4°C for up to 28 days	Cadmium reduction, Automated	353.2, Rev. 2.0 (1993)	4500-NO ₃ ⁻ F-2000	D3867-04 (A)	I-2545-90. ⁵¹
	Automated hydrazine		4500-NO ₃ ⁻ H-2000		

Parameter/Preservative/ Holding Time/Bottle Type	Methodology	EPA	Standard methods	ASTM	USGS/AOAC/ Other
200-mL; plastic or glass	Reduction/Colorimetric				See footnote. ⁶²
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997)	4110 B-2000 or C-2000	D4327-03	993.30. ³
	CIE/UV		4140 B-1997	D6508-00(05)	D6508, Rev. 2. ⁵⁴
40. Nitrite (as N), mg/L	Spectrophotometric: Manual		4500-NO ₂ ⁻ B-2000		See footnote. ²⁵
Store at 4°C for up to 48 hours	Automated (Diazotization)				I-4540-85 ² , See footnote. ⁶²
100-mL; plastic or glass	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993)	4500-NO ₃ ⁻ F-2000	D3867-04 (A)	I-4545-85. ²
	Manual (*bypass cadmium reduction)		4500-NO ₃ ⁻ E-2000	D3867-04 (B)	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1-1, Rev 1.0 (1997)	4110 B-2000 or C-2000	D4327-03	993.30. ³
	CIE/UV		4140 B-1997	D6508-00(05)	D6508, Rev. 2. ⁵⁴
43. Organic nitrogen (as N), mg/L Add H ₂ SO ₄ to pH < 2, store at 4°C for up to 28 days	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)				
50. Phosphorus—Total, mg/L	Digestion ²⁰ , followed by any of the following:		4500-P B(5)-1999		973.55. ³
Add H ₂ SO ₄ to pH < 2, analyse ASAP or store at 40C for up to 28 days	Manual	365.3 ¹ (Issued 1978)	4500-P E-1999	D515-88 (A)	
	Automated ascorbic acid reduction	365.1 Rev. 2.0 (1993)	4500-P F-1999, G- 1999, H-1999		973.56 ³ , I-4600- 85. ²
100-mL; plastic or glass	ICP/AES ^{4, 36}	200.7, Rev. 4.4 (1994)	3120 B-1999		I-4471-97. ⁵⁰
	Semi-automated block digester (TKP digestion)	365.4 ¹ (Issued 1974)		D515-88 (B)	I-4610-91. ⁴⁸

Parameter/Preservative/ Holding Time/Bottle Type	Methodology	EPA	Standard methods	ASTM	USGS/AOAC/ Other
Total Nitrogen, (as N), mg/l	Lachat 10-107-04-4 ^A				
Add H ₂ SO ₄ to pH < 2, store at 4°C. If frozen it can be stored for up to 30 days					
<i>100-mL; plastic or glass</i>					

Notes/Footnotes

^A Approved for use by the Colorado Water Quality Control Division

¹ Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020. Revised March 1983 and 1979, where applicable. U.S. EPA.

² Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Book 5, Chapter A1., unless otherwise stated. 1989. USGS.

³ Official Methods of Analysis of the Association of Official Analytical Chemists, Methods Manual, Sixteenth Edition, 4th Revision, 1998. AOAC International.

⁴ For the determination of total metals (which are equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. The approved total recoverable digestion is described as Method 200.2 in Supplement I of “Methods for the Determination of Metals in Environmental Samples” EPA/600R-94/111, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP-AES, and ICP-MS) use EPA Method 200.2 or an approved alternate procedure (e.g., CEM microwave digestion, which may be used with certain analytes as indicated in Table IB); the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as “total” metals.

⁵ Copper sulfate or other catalysts that have been found suitable may be used in place of mercuric sulfate.

⁷ Industrial Method Number 379-75 WE Ammonia, Automated Electrode Method, Technicon Auto Analyzer II. February 19, 1976. Bran & Luebbe Analyzing Technologies Inc.

⁸ The approved method is that cited in Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1. 1979. USGS.

⁹ American National Standard on Photographic Processing Effluents. April 2, 1975. American National Standards Institute.

²⁰ When using a method with block digestion, this treatment is not required.

²⁵ Method 8507, Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater. 1979. Hach Company.

³⁰ The use of EDTA decreases method sensitivity. Analysts may omit EDTA or replace with another suitable complexing reagent provided that all method specified quality control acceptance criteria are met.

³⁶ Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals. April 16, 1992. CEM Corporation

³⁹ Method PAI-DK01, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Titrimetric Detection. Revised December 22, 1994. OI Analytical.

⁴⁰ Method PAI-DK02, Nitrogen, Total Kjeldahl, Block Digestion, Steam Distillation, Colorimetric Detection. Revised December 22, 1994. OI Analytical.

⁴¹ Method PAI-DK03, Nitrogen, Total Kjeldahl, Block Digestion, Automated FIA Gas Diffusion. Revised December 22, 1994. OI Analytical.

⁴⁵ Open File Report 00-170, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonium Plus Organic Nitrogen by a Kjeldahl Digestion Method and an Automated Photometric Finish that Includes Digest Cleanup by Gas Diffusion. 2000. USGS.

⁴⁸ Open File Report 92-146, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis. 1992. USGS.

⁵⁰ Open File Report 98-165, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry. 1998. USGS.

⁵¹ Open File Report 93-125, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments. 1993. USGS.

⁵⁴ Method D6508, Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte. December 2000. Waters Corp.

⁶⁰ Analysts should be aware that pH optima and chromophore absorption maxima might differ when phenol is replaced by a substituted phenol as the color reagent in Berthelot Reaction (“phenol-hypochlorite reaction”) colorimetric ammonium determination methods. For example when phenol is used as the color reagent, pH optimum and wavelength of maximum absorbance are about 11.5 and 635 nm, respectively—see, Patton, C.J. and S.R. Crouch. March 1977. *Anal. Chem.* 49:464-469. These reaction parameters increase to pH > 12.6 and 665 nm when salicylate is used as the color reagent—see, Krom, M.D. April 1980. *The Analyst* 105:305-316.

⁶² Easy (1-Reagent) Nitrate Method, Revision November 12, 2011. Craig Chinchilla.

Appendix C: Electronic Data Format

Format for submission to Data Sharing Network is set by DSN. The Division's format is an interactive Excel entry form that is pre-populated with 2013 Regulation 85 monitoring data for each discharger. Every discharger should have received a data entry form in an email from the Division. If this form was not received contact the Division at CDPHE_nutrients@state.co.us.

Appendix D: Example Chain of Custody

Agency: _____

Agency Address: _____

CHAIN OF CUSTODY RECORD

CDPS PERMIT #			SAMPLERS (SIGNATURE)			
LOCATION	DATE	TIME	SAMPLE TYPE (Grab/Composite)	SAMPLE TIME	# OF CONTAINERS	ANALYSIS REQUESTED
RELINQUISHED BY: (SIGNATURE)			RECEIVED BY: (SIGNATURE)			DATE/TIME
RELINQUISHED BY: (SIGNATURE)			RECEIVED BY: (SIGNATURE)			DATE/TIME
RELINQUISHED BY: (SIGNATURE)			RECEIVED BY: (SIGNATURE)			DATE/TIME
DISPATCHED BY: (SIGNATURE)		DATE/TIME	RECEIVED FOR LABORATORY BY:			DATE/TIME
METHOD OF SHIPMENT:						
RELINQUISHED BY: (SIGNATURE)			RECEIVED BY: (SIGNATURE)			DATE/TIME
RELINQUISHED BY: (SIGNATURE)			RECEIVED BY: (SIGNATURE)			DATE/TIME
DISTRIBUTION: ORIGINAL-ACCOMPANY SHIPMENT 1 COPY-COORDINATOR						