



**SAMPLING AND ANALYSIS PLAN**

**A GUIDE FOR BIOSOLIDS SAMPLING**

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Fire Mountain Farms, Inc.  
Sample Analysis Plan  
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BIOSOLIDS SAMPLING

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## 1.0 Introduction

An important part of the biosolids program is based upon valid analytical data derived from relatively small samples. The collection of a sample and its proper preservation during shipment is fundamental to obtaining reliable analytical results.

The concentration of nutrients, pathogens, and pollutants in biosolids are variable. In addition, pathogenic organisms are both time and temperature sensitive. Establishing a written protocol is important in order to collect samples that are both representative and consistent. In accordance with WAC 173-308-140 (1), all sampling that is conducted must be representative of the biosolids that are to be land applied.

Analysis of potential pollutants (so-called 503 metals) and pathogens provide the basis for establishing Class B biosolids. Nutrient concentrations are used to determine agronomic rates when biosolids are land applied. The analysis of biosolids will follow the methods outlined in WAC 173-308-140. Biosolids sampling frequency is set forth in WAC 173-308-150 see table 1. Samples shall be tested for the pollutants in section WAC 173-308-160. The pollutants that shall be tested are: arsenic, cadmium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc.

In addition to sampling biosolids are collected at land application sites to provide important crop nutrient data. The nitrogen status of the soil is combined with the nitrogen of the biosolids and the predicted crop uptake to develop the agronomic rates at which the biosolids are to be applied to the land. Accurate assessment of soil nitrogen is dependent on good sampling techniques.

This sampling and analysis plan (SAP) will describe the processes involved with sampling biosolids at Fire Mountain Farms, Inc. The intent is to be complete and concise so that sample collection, preservation, and shipment to a laboratory for appropriate analysis may be performed by personnel with little or no assistance outside this document.

## 2.0 Laboratory Designation

When Fire Mountain Farms chooses a laboratory to perform the required analysis of biosolids samples they will determine and verify some important aspects of the analysis program:

1. Ensure that the laboratory is accredited by the State of Washington. Fire Mountain Farms, Inc. personnel will contact the laboratory prior to collecting biosolid samples to ensure that the lab is accredited by the State of Washington for Solids and Chemical Materials matrix. Fire Mountain Farms, Inc. personnel may also use a website to search for accredited laboratories (<https://fortress.wa.gov/ecy/laboratorysearch/>).

2. Determine that the required analyses can be performed by the laboratory. Fire Mountain Farms, Inc. personnel will confirm with the lab to make sure that all requested analyses can be performed. Fire Mountain Farms, Inc. personnel will check to confirm that the lab is approved by Washington State Department of Ecology.
3. Ensure that required turn-around times can be met by the laboratory. Fire Mountain Farms, Inc. will contact the laboratory to make certain that the requested turn-around-times for submitted analyses can be met by the laboratory. If turn-around-time can't be met, Fire Mountain Farms, Inc. may search for another laboratory that meets accreditation criteria.
4. Make sure that the laboratory understands how to report the results (e.g. dry-weight basis, mg/kg, etc.). Fire Mountain Farms, Inc. will direct the laboratory as to how the desired layout of results reports should appear. Analysis results will be reported on a dry weight basis. Due to variability of percent solids on some projects dry weight basis will allow Fire Mountain Farms staff to make adjustments to application rates as needed.

Fire Mountain Farms, Inc. may submit a copy of this SAP to the reporting lab so that the required analyses and sample collection protocol may be on record for them to review.

Due to the large geographic region in which Fire Mountain Farms, Inc. operates, specific laboratories used for analysis of biosolids are not listed. Fire Mountain Farms, Inc. will ensure that all biosolids analyses are performed by a Washington State accredited laboratory, as described in *General Permit for Biosolids Management* (General Permit).

### **3.0 Biosolids Sampling Protocol**

A number of tools and personal protection equipment will be required to complete the task aseptically so as to avoid contamination of one's self or the sample. Collected samples will be placed in sample bottles provided by laboratory and chilled to 4<sup>0</sup> C using ice, blue ice, or dry ice. All equipment that is used to collect and prepare samples must be prepared so that it does not contaminate or react with the material being sampled. Contamination can arise if equipment is improperly cleaned or is made of materials that are released into the sample. (Galvanized or chrome-plated implements must be avoided.) Relatively inert materials such as Teflon, glass, or stainless steel are typically used for sampling equipment or containers. In certain situations, plastic, non-stainless steel, or aluminum sampling equipment can be used in place of the preferred materials. For example, if a sample is collected for metals analysis, a plastic sampling device or container is acceptable. Sampling equipment needs to be chosen based on the analysis being performed as well as the consistency of the material being sampled.

**Examples of Fire Mountain Farms, Inc. equipment are as follows:** stainless steel spatula, disposable plastic spatula, shovel, custom built sludge judge, sampling tube, soil sampling probe or auger and or other equipment approved by Department Of Ecology. Samples and sample equipment for Fire Mountain Farms, Inc. site location vary based on the type of samples being taken and type of site. Prior to collecting samples Fire Mountain Farms personnel will have to determine what tools will be needed. Some examples are listed below.

**Mixed during Staging:** Fire Mountain Farms personnel will use a shovel, spatula, sampling tube, soil sample probe, or other equipment approved by the Department of Ecology to take the sample, sample containers with labels filled out at the office except for the time of sample, a cooler, and the chain of custody paperwork for the lab that will be providing the analysis. Some personal equipment: latex or nitrile gloves.

**Sample:** All samples are required to have sample container with a label filled with the sample names (time that the sample is taken is placed on label on site as soon as the sample is taken) and a chain of custody provided by the lab that will be performing the analysis(chain of custody will be filled out prior with all types of samples that are being taken, names of samples, what type of material that is being collect for sampling, (time of sample to added after sample is taken). 24 hour notice to the lab is generally required before taking samples.

For each fecal coliform test you will need seven 100ml or larger sample containers per test. A fecal coliform tests need to be chilled to 4°C directly after sample is taken and delivered to the lab within 6 hours of the sample being collected.

For a vector attraction reduction sample (VARs) you will need one 1 liter sample container. This sample is cooled and shipped overnight up to the lab for analysis or driven up to lab same day; however the lab needs to be notified 24hours in advance. WAC 173-308-180

For TKN, Ammonia, Nitrate, and Potential Pollutants (503 metals): one 500ml sample is taken. The time limit for analysis is up to 6 months. Fire Mountain Farms normally delivers samples same day but these sample could be shipped in a cooler within a few day. WAC 173-308-160 & WAC 173- 308-205

**Cleaning and locations of equipment:** all equipment can be located in the Fire Mountain Farms, office. After collecting the samples, the equipment needs to be cleaned on site with soap and water then dried and wrapped in an inert material such as aluminum foil or plastic to protect it until the next use. Tools are returned to operations office.

### 3.1 Tools Required for Sample Collection

- Prior to anyone new using sample equipment, experienced Fire Mountain Farms, Inc. personnel will demonstrate and train on how to use the equipment and fill out the required paperwork for the lab (s) and Fire Mountain Farms, Inc. records.
- Latex or Nitrile gloves (be sure to have plenty on hand)
- Sample containers will be obtained from laboratory doing the analysis. Sample containers from our stock can be obtained from Fire Mountain Farms, Inc. office personnel, for fecal coliform samples Fire Mountain Farms, Inc. will take seven 100ml sample. For vector attraction reduction samples (VARs) we will take one 1 liter sample and for “503” metals we will take one 500ml sample. Contact the laboratory at least two weeks prior to sampling event to request sample containers if none are on hand)
- Ice chest with ice, “blue ice”, or dry ice for transporting the samples.
- Shipping containers (may be the ice chest or Styrofoam –type container)
- Sharpie® pens, ink pens, labels for sample containers
- Chain of Custody forms. For the lab (s) to which the sample will be delivered.
- Suitable sample collection equipment (depends on type of biosolids collected). Shovel, stainless steel spatula or disposable plastic spatula for dewatered biosolids or custom built “sludge judge” for sampling lagoon storage units
- Stainless steel bowl or suitable container to composite samples

### 3.2 Sampling for Nutrients, Metals, Total & Volatile Solids

- 1) Ensure that you have the appropriate labels. A description label includes project name, sample name, date, analysis, preservative and a comment section on it them. These sample labels need to be on the containers **before** samples are collected. After the sample is collected write in the time that the sample was collected on the label that is on your sample container. These labels shall be picked up from Fire Mountain Farms Inc. office personnel. Labeling is the same for all types of samples, ill-regardless of percent solid or source.
- 2) Ensure that you have the appropriate containers. The sample containers are provided by the lab (s) prior to use and more can be picked up when samples are delivered. Generally, sample containers are made of glass or plastic because these materials are relatively inert and easily cleaned. Sample containers come in several sizes. For Fire Mountain Farms, Inc. the container sizes are generally 100, 250, 500 milliliters or 1 liter. For vector attraction reduction samples (VARs) we will take one 1 liter sample and for “503” metals we will take one 500ml sample. These volumes should be sufficient but if the lab performing the analysis requests different volumes you will need to comply with their request. A special case, for example, would be

using a 1 gallon container for samples collected to determine the appropriate polymer for use in de-watering.

- 3) Ensure that you have the appropriate Chain of custody form for the lab you intend on utilizing for sample analysis. This form can be obtained in the Fire Mountain Farms, Inc. office file cabinet in the first drawer or from the office personnel. These can also be picked up from the lab (s) prior to samples being taken or downloaded from the lab (s) internet site. These should be filled out with Fire Mountain Farms, Inc. information, sample identification, types of test you want per sample, and after the sample is taken, the time that the sample was collected.
- 4) Put on your latex or nitrile gloves that are found in the Fire Mountain Farms, Inc. office.
- 5) Map out where you plan to collect biosolids samples using the map provided by Fire Mountain Farms, Inc. office personnel. 10-15 samples should be taken that are representative of current volume of biosolids.

Use appropriate equipment for the type of biosolids (e.g. liquid or solid) you are collecting. The equipment needed is to be determined before sampling: stainless steel spatula, disposable plastic spatula, shovel, custom built sludge judge, sampling tube, soil sampling probe, auger, or equipment approved by DOE. Each sample location varies on the types of equipment that Fire Mountain Farms, personnel will use. Collect and place 10-15 small (the number of samples collected varies due to size of field/ lagoon/bunker being sampled,) separate, grab-samples of biosolids into a stainless bowl or suitable container (an example of a suitable container would be a clean plastic bucket when collecting liquid samples from a lagoon. Prior to someone new collecting samples, Fire Mountain Farms, Inc. experienced personnel will train and demonstrate how to use all sampling equipment.

Some examples are:

**Mixed during Staging:** Fire Mountain Farms personnel will use a shovel, spatula, sampling tube, soil sample probe, or other equipment approved by the Department of Ecology to take the sample, sample containers with labels filled out at the office except for the time of sample, a cooler, and the chain of custody paperwork for the lab that will be providing the analysis. Some personal equipment: latex or nitrile gloves.

**Fields:** Fire Mountain Farms personnel will use stainless steel spatula, disposable plastic spatula, shovel, sampling tube, soil sample probe, mixing chamber, or other equipment approved by Department of Ecology to collect the samples. Sample containers with labels filled out at the office except for the time of sample, a cooler, and the chain of custody paperwork for the lab that will be performing the analysis. Some personal equipment: latex or nitrile gloves.

**Cleaning and locations of equipment:** all equipment can be located in the Fire Mountain Farms, office. After collecting the samples, the equipment needs to be cleaned on site with soap and water then dried and wrapped in an inert material such as aluminum foil or plastic to protect it until the next use.

- 6) Mix the grab samples together thoroughly in the composite sample container. The number of samples added to the composite mix needs to be representative of size of area and distribution of solids in the storage unit with a minimum of 10 sample. The total sampled amount should be more than the amount needed to send to the lab.
- 7) Once the grab samples have been thoroughly mixed in the composite bowl or container, take a portion of the total and place it in the lab supplied container. Fill the container so that the lab has enough biosolids for the analysis. 500ml to 1 liter samples are sufficient for nutrients, % solids and metals but volatile solids reduction tests will require a 1 liter sample.
- 8) Place the sample into a travel container to begin preservation (see Table-1 for preservation). (Note: VRS samples should be maintained at ambient temperature.
- 9) After collecting the samples, the equipment needs to be cleaned on site with soap and water then dried and wrapped in an inert material such as aluminum foil or plastic to protect it until the next use. For storage, bring back the equipment to the Fire Mountain Farms, Inc. office to be placed in its appropriate storage area until needed for another sampling.

### 3.3 Sampling for Fecal Coliform - 7 Samples Method (Alternative 1) - WAC 173-308-170

- 1) Ensure that you have the appropriate labels and 100ml sample containers. A description label includes project name, sample name, date, analysis, preservative and a comment section on it them. These sample labels need to be on the containers **before** samples are collected. After the sample is collected write in the time that the sample was collected on the label that is on your sample container. These labels and sample containers shall be picked up from Fire Mountain Farms, Inc. office personnel. The sample containers are provided by the laboratory prior to use and more can be picked up when samples are delivered.
- 2) Put on nitrile rubber gloves.
- 3) Mapping out where you plan to collect biosolids samples from. This will be done based on what location and type of sample that is being taken. **An example is:** Office personnel will provide a map of the lagoon. If the lagoon is currently operating with an agi-pump a sample can be taken out of the line at the sampling point. If there is no agi-pump operating personnel will take a sludge jug, sample tube, or other approved sample equipment by Department Of Ecology. Samples are taken in containers ranging from 100ml to 1 liter plastic or glass containers approved by

Department Of Ecology and the Lab performing the testing. Samples should be taken that are representative of current volume of biosolids.

- 4) Using appropriate equipment for type of biosolids.  
Examples are: stainless steel spatula, disposable plastic spatula, shovel, custom built sludge judge, agi-pump lines, soil sampling probe, auger, and other approved equipment by Department Of Ecology. Each sample location varies on the types of equipment that Fire Mountain Farms, personnel will use.  
Some examples are:  
**Mixed during Staging:** Fire Mountain Farms personnel will use a shovel, spatula, soil sampling probe, or other Department of Ecology approved equipment to take the sample. Some personal safety equipment: latex or nitrile gloves.  
**Fields:** Fire Mountain Farms personnel can use stainless steel spatula, disposable plastic spatula, shovel, soil sampling probe, auger, or other Department Of Ecology approved equipment to collect samples. Collect a single sample and place it in the lab-supplied container Examples are: 100ml containers to 1 liter containers plastic or glass. Some personal equipment: latex or nitrile gloves.
- 5) **Immediately place the sample on ice** and into the shipping container to begin preservation (see Table-1 for preservation).
- 6) Seven individual samples need to be collected for fecal coliform analysis. Fecal coliform samples shall arrive at the accredited laboratory conducting the analysis **within 6 hours of collection.**

#### 4.0 Shipping Samples

- 1) Complete the chain of custody form. **Be sure that the chain of custody form is accurate!** This is an area where misidentification is common and a careful “second-check” will help greatly in keeping paperwork and sample identification in order.
- 2) Ensure lids are sealed tightly. Pack samples into the shipping container. Place regular ice into sealable plastic bags and pack into shipping container around the samples. If it’s hot weather, dry-ice may be necessary to properly preserve the sample during shipment.
- 3) If there is space in the shipping container that allows the sample containers to easily move around, fill the void with packing material.
- 4) Place the completed Chain of custody form into a sealable plastic bag, seal, and place it into the shipping container. **This is important!** It keeps the paperwork dry and legible when it arrives at the lab. Tape the container securely shut with packing tape. If you cannot drop the package from 3 feet onto the floor and be confident that the contents will not be damaged, it is improperly packed.
- 5) Transport samples to shipping facility (e.g. UPS, FedEx, etc.). Due to short hold times, it will normally be necessary for FMF staff to directly deliver samples to

laboratory when testing for Fecal Coliform bacteria and Vector Attraction Reduction Samples (VARs). (See table 5.1 below)

## 5.0 Biosolids Analysis

Ensure the chain of custody lists the appropriate constituents to be analyzed. The following list can be utilized to double-check for completeness of the chain of custody form. If there are mistakes or omissions, the analysis will be wrong or insufficient. If a constituent is not listed on the chain of custody form received from the laboratory, or you are uncertain about some detail, **CALL THE LAB AND CONFIRM BEFORE SHIPPING.**

**Table 5.1-Biosolids Analytical Methods and Holding Time**

Constituent	Analysis Method	Preservation	Hold-Time
Arsenic	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Cadmium	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Copper	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Lead	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Molybdenum	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Nickel	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Selenium	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Zinc	SW-846 Method 6010	Cool to ~4° Celsius	6 months
Mercury	SW-846 Method 7471	Cool to ~4° Celsius	6 months
Vector Attraction Reduction	EPA/625/R-92/013	Cool to ~4° Celsius	24 Hours
Total Kjeldahl Nitrogen	SM 4500- N <sub>org</sub> C <u>or</u> EPA PAI-DK 01	Cool to 4° Celsius	28 days
Nitrate – N	EPA 300.0 or 353.2	Cool to 4° Celsius	28 days
Ammonia – N	SM4500-NH <sub>3</sub>	Cool to 4° Celsius	28 days
Fecal Coliform	SM 9221 E <u>or</u> EPA Method 1680 <u>or</u> 1681	Cool to 4° Celsius	6 HOURS TO LAB, 24 HOURS FOR ANALYSIS
Total Solids	SM 2540 G	Cool to 4° Celsius	7 days

## 5.1 Biosolids analysis evaluation

When Fire Mountain Farms, Inc. receives analytical results they will be evaluated to determine if the biosolids will meet the class B requirements for land application. Special attention will be given to common errors such as metals results with detection levels higher than those listed in table 5.2- Biosolids limits for potential pollutants listed below and results provided in wet basis rather than dry weight basis. Analysis will also be reviewed and evaluated to determine if results are within and not pushing close to (table 3) limits. As a general rule we will take action anytime a result is within 75% of the table 3

limit as shown in table 5.2 below. The first action when analytical results confirm levels outside limits would require consultation with Ecology to determine appropriate steps to take.

Below in table 5.2 are limits for potential pollutants in biosolids.

**Table 5.2-Biosolids limits for potential pollutants**

		Concentration Limit	
Element	Symbol	("503" Table 3)	("503" Table 1) (Ceiling Limit)
		mg/kg	mg/kg
Arsenic	As	41	75
Cadmium	Cd	39	85
Copper	Cu	1500	4300
Lead	Pb	300	840
Mercury	Hg	17	57
Molybdenum	Mo	*	71
Nickel	Ni	420	420
Selenium	Se	100	100
Zinc	Zn	2800	7500

### 67.0 Biosolids Scenarios

The Washington State Department of Ecology (Ecology) allows the mixing of non-exceptional quality (non-EQ) biosolids products produced by different sources. However, when this occurs the mixer is viewed as a person who prepares a *material derived from biosolids*. A material derived from biosolids is considered to be a new biosolids product that must meet all applicable quality standards and management requirements in the state biosolids rule (*Chapter 173-308 WAC, Biosolids Management*). Current scenario for Biosolids that would need to be re sampled is if materials were accidentally mixed during staging. Due to trucks dumping not in their assigned spot.

#### Alternatives

<b>Alternative 1</b>	Fecal coliform are less than 2,000,000 most probable number or 2,000,000 colony-forming units per gram of total solids, based on a geometric means of seven samples.
<b>Alternative 2</b>	Use a process to significantly reduce pathogens (PSRP)

<b>Alternative 3</b>	Use a process determined by the state or the EPA to be equivalent to a process to significantly reduce pathogens.
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***Frequency of Sampling***

The mixing facility will be required to sample in accordance with the frequency requirements in WAC 173-308-150. The frequency of sampling is based on the dry weight tonnage of biosolids applied to the land in a given year. The minimum frequency of monitoring applies to pollutants, pathogen reduction, and vector attraction reduction. The minimum frequency of monitoring is:

**Biosolids Sampling Frequency**

Metric dry tons (U.S. tons) per 365-day period	Frequency
Greater than zero but less than 290 (320)	Once per year
Equal to or greater than 290 (320) but less than 1,500 (1,653)	Once per quarter (4 times per year)
Equal to or greater than 1,500 (1,653) but less than 15,000 (16,535)	Once per 60 days (6 times per year)
Equal to or greater than 15,000 (16,535)	Once per month (12 time per year)

**Method of Meeting Vector Attraction Reduction Standards**

<b>1</b>	Biosolids digestion processes with greater than 38% volatile solids reduction
<b>2</b>	Test end-product of anaerobic digestion process. Forty-day anaerobic test at 30-37°C. Acceptable stabilization if less than 17% volatile solids reduction occurs during the test.
<b>3</b>	Test end-product of aerobic digestion process having less than 2% solids. Thirty-day aerobic test at 20°C. Acceptable stabilizations if less than 15% volatile solids reduction occurs during the test.
<b>4</b>	<b>Facilities with aerobic digestion.</b> Specific oxygen uptake rate (SOUR) testing using end-product of digestion process. Acceptable stabilization if uptake is less than 1.5 mg oxygen per g total solids per hour at 20°C
<b>5</b>	<b>Time/ temperature requirement for composting:</b> Fourteen days residence time at temperatures greater than 40°C, with average temperature greater than 45°C
<b>6</b>	<b>High pH stabilization.</b> Biosolids pH above 12 for 2 hours and greater than 11.5 for 22 hours.

7	<b>Treatment by drying.</b> Not to include unsterilized primary wastewater solids. Total solids content greater than 75% before mixing, with other material.
8	<b>Treatment by drying.</b> Can include unstabilized primary wastewater solids. Total solids greater than 90% before mixing with other materials.
9	<b>Barrier process.</b> Injection into soil. No biosolids on soil surface 1 hour after application. For Class A biosolids, injection must occur within 8 hours of discharge from the pathogen reducing process. See WAC 173-308-210, 220,230,240(3)
10	<b>Barrier process.</b> Soil incorporation by tillage. Soil incorporation by tillage within 6 hours of application. For Class A biosolids, application must occur within 8 hours of discharge from the pathogen reducing process. See WAC 173-308-210,
11	<b>Septage only.</b> High pH treatment before land application. Acceptable stabilization if pH is greater than 12 for 30 minutes. See WAC 173-308-270 (4)