

# PFAS Biosolids / Sludge Field Sampling & Cross Contamination



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**Water Division - New Hampshire Department of Environmental Services (NHDES)**

**2020 NEWMOA Webinar Series**  
**June 23<sup>rd</sup>, 2020**  
**1:30pm - 3:00pm**

# Outline

- ▶ **NHDES Residuals Management Section**
- ▶ **NHDES Sludge Management Rules: Sludge Quality Certification Program (SQC)**
- ▶ **NHDES SQC PFAS Investigations**
  - ▶ **NEIWPC and NEBRA Guidance: Sampling and Analysis of PFAS in Biosolids and Associated Media**
  - ▶ **Quality of sample equipment cleaning & sampling**
- ▶ **2017 – 2019 NH SQC PFAS data review**



# Residuals Management Section

## NHDES

- ▶ Water Division
  - ▶ Wastewater Engineering Bureau
    - ▶ Residuals Management Section

Anthony  
Drouin  
**Administrator**  
*Sludge Quality  
Certification  
Permitting*

Judith  
Sears-Houston  
**Permitting &  
Enforcement  
Engineer**  
*Site and Facility  
Permitting*

Vacant  
**Sludge & Septage  
Coordinator**  
*Training and  
Outreach*

Timothy  
Sweatt  
**Inspector**  
*Sludge & Septage  
Hauler Permitting*

# Applicable State Rules for Residuals Management Options



- ▶ Land Application - Env-Wq 800/Env-Wq 1600/RSA 485-A
- ▶ Solid Waste - Env-Sw 100 through 2100, RSA 149-M
- ▶ Incineration - Env-A 600, 40 CFR Part 60, Subpart O
- ▶ Federal Regulations - 40 CFR Part 503



# Sludge & Septage Management:

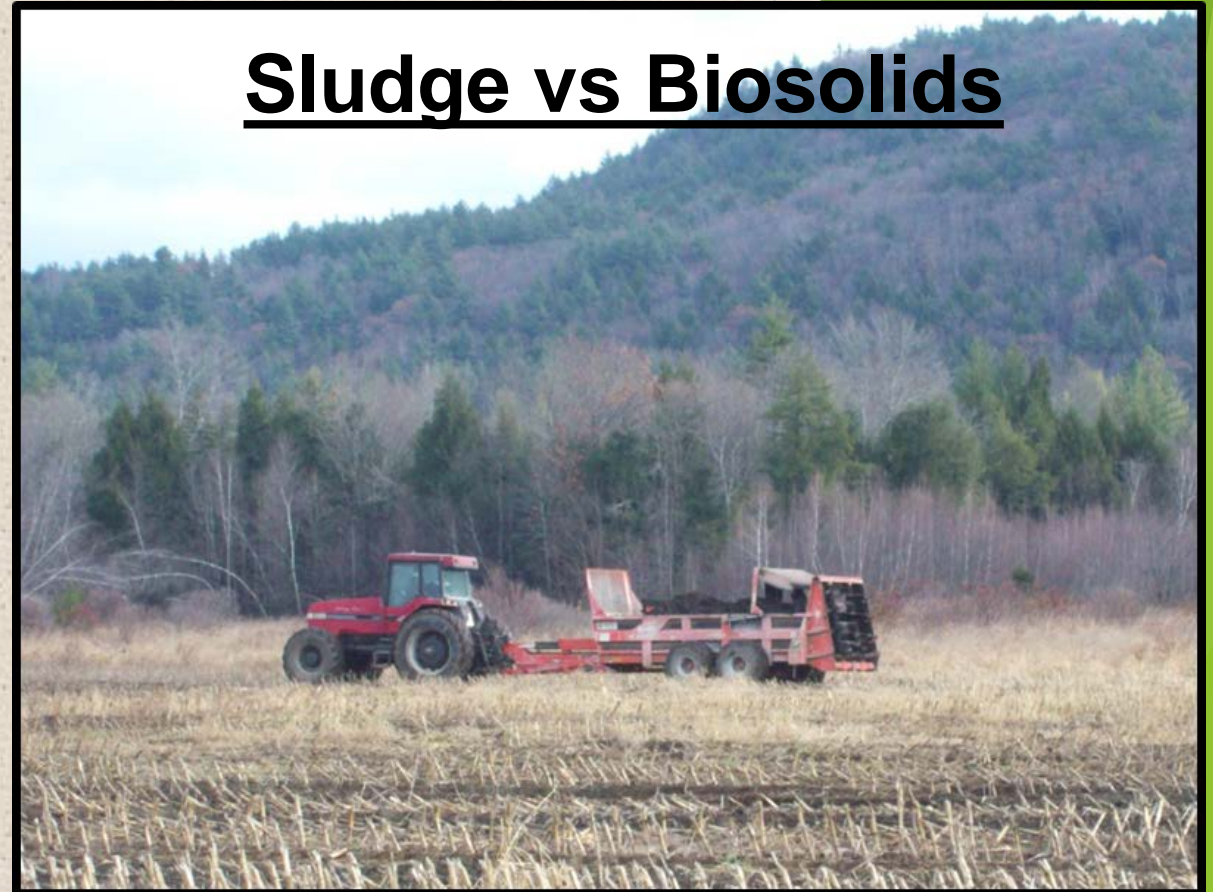
- Hauling
- Residuals Quality
- Sites/Land Application
- Facilities
- Recordkeeping
- Reporting to NHDES



# Sludge Quality Certification

- Basic information
- Industrial pretreatment information
- Quantity
- Pathogen & vector attraction reduction options
  - Class A & Class B biosolids
    - **P**rocess to **S**ignificantly **R**educe **P**athogens: Class B
    - **P**rocess to **F**urther **R**educe **P**athogens: Class A
  - Short Paper Fiber
  - Drinking Water Residuals
- Historical & current quality data

## Sludge vs Biosolids





# Sludge Quality Certification

- Annual Testing (frequency of testing based on volume generated)
- Recordkeeping
- Annual Report to NHDES by last business day in **January**, each year
- Renew certificate every 5 years





# Sludge Quality Certification

**Beneficial Use is the utilization of the nutrients and organic matter from the biosolids for the agronomic need as long as it does not pose a significant threat to human health or environment. Beneficial use applies to agricultural, forest, and land reclamation management practices**

- Bulk biosolids must obtain an SQC to be distributed in NH
- Annual soil test determine crops nutrient demand supplied from biosolids (UNH BMP)
- Class B land application must obtain a site permit through RMS
- Concentration limits and screening standards set for VOC, SVOC, PCB's, Dioxin, & Metals – **168 analytes**





# 2017 Initial Sludge PFAS Investigation



## State of New Hampshire



DEPARTMENT OF ENVIRONMENTAL SERVICES  
WASTEWATER ENGINEERING BUREAU  
RESIDUALS MANAGEMENT SECTION  
29 HAZEN DRIVE, P.O. BOX 95  
CONCORD, NEW HAMPSHIRE 03301



### FACILITY/SITE INSPECTION REPORT

Inspection Report #: 1705

Type (Sludge/Septage): Sludge sampling

Name/Location: Peterborough WWTF

Permit No: N/A

Date of Inspection: April 19, 2017

Inspectors: Ray Gordon &  
Judy Sears Houston

Person(s) On-Site: Peterborough Operators – Nate Brown & Jamie Jarest

#### Sampling Report

On Wednesday, April 19, 2017, staff of the Residuals Management Section (RMS) collected a compost sample from the dewatered sludge located in 11 "geobags" currently at the Peterborough Wastewater Treatment Facility (WWTF). These geobags contain sludge from the partial closure of Lagoon #1 from the previous two years. The bags were placed on poly and surrounded by an earthen/gravel berm by the Town, in accordance with the DES-approved Lagoon Closure Plan. As the Town is trying to decide which proposed alternative to implement for the disposal of the dewatered sludge, RMS agreed to sample the sludge from the geobags as part of the annual sludge generator sampling program, per RSA 485-A:4, XVI-c.(a). This sampling also included the collection of a sample for polyfluorinated compounds (PFCs) testing, as part of the Department's ongoing efforts to identify impacts of PFC's and related compounds on the environment.

RMS staff arrived at the Peterborough WWTF at about 11:30 AM. RMS staff cleaned all sampling equipment from the previous sampling location in the Town's WWTF laboratory. The steel trowel, soil probe and bucket were first cleaned with the facility's hand soap ("Carex 3000 Green Liquid Soap, General Purpose, SKU 48020" by Georgia Pacific) then rinsed 3 times with DES laboratory deionized water, which has been analyzed and shown to not contained PFCs. The clean soil probe was dried with paper towels and wrapped with paper towels then with tinfoil, to keep it clean during transport to the geobag storage location.

At the dewatering geobag location, RMS noted there where two different sized bags filled with dewatered sludge. The geobags had been placed right up against each other, with no measureable space between the bags. Two different sized geobags were measured and counted by RMS staff:

- 9 longer bags each approximately = 27 ft. wide x 198 ft. long x 1.5 ft. thick; and
- 2 shorter bags each approximately = 27 ft. wide x 170 ft. long x 1.5 ft. thick.

The 11 geobags totaled approximately 3,183 cy of dewatered sludge. The Town operators indicated that the geobags were originally approximately 6 feet high when wet sludge was first pumped into them; significant dewatering of the sludge is apparent during this inspection. (Refer to **Photos #1, #2, and #3**, below, and field notebook sketch, for approximate configuration of the geobags.)

- 12 SQC and **Non SQC** permit holders tested in 2017
- Sampling conducted by NHDES Residuals Management Section
- Samples analyzed by Test America in Sacramento, California and Maxxam in Mississauga, Ontario (2 samples)
- Class A & Class B biosolids, WWTF sludge, and short paper fiber from paper mills were sampled
- 9 PFAS compounds were analyzed



(ng/g) (ppb)	# of Detections	% of Detections	Min.	Max.	Mean	Standard Deviation	Average Deviation
PFBA	4.0	33.3	0.5	12.0	4.1	5.3	3.9
PFPeA	5.0	41.7	0.8	27.0	7.0	11.2	8.0
PFHxA	9.0	75.0	1.3	73.0	11.6	23.2	13.7
PFHpA	5.0	41.7	0.5	4.6	2.0	1.7	1.4
PFOA	9.0	75.0	1.1	13.0	4.1	3.7	2.6
PFNA	8.0	66.7	1.5	3.6	2.8	0.8	0.7
PFBS	2.0	16.7	0.3	5.2	2.7	2.7	5.8
PFHxS	6.0	50.0	0.5	73.0	14.0	28.9	19.7
PFOS	10.0	83.3	7.2	390.0	54.5	118.4	67.1

## 2018 Initial Sludge PFAS Investigation

- 16 Sludge Quality Certificate Holders tested in 2018
- Sampling conducted by NHDES Residuals Management Section
- Samples analyzed by Test America in Sacramento, California
- Class A & Class B biosolids, WWTF sludge, drinking water treatment residuals, and short paper fiber from paper mills were sampled
- 9 PFAS Compounds were analyzed



(ng/g) (ppb)	# of Detections	% of Detections	Min.	Max.	Mean	Standard Deviation	Average Deviation
PFBA	11.0	68.8	0.2	150.0	16.0	44.6	24.4
PFPeA	9.0	56.3	0.5	55.0	9.9	17.6	10.9
PFHxA	11.0	68.8	1.7	630.0	73.4	188.4	111.5
PFHpA	10.0	62.5	0.1	10.0	1.6	3.1	1.9
PFOA	13.0	81.3	0.4	19.0	4.2	6.3	4.8
PFNA	15.0	93.8	0.1	4.0	1.2	1.3	1.0
PFBS	5.0	31.3	0.2	37.0	11.4	15.1	10.5
PFHxS	9.0	56.3	0.1	2.7	1.0	0.8	0.6
PFOS	15.0	93.8	1.7	29.0	12.0	9.3	7.6



VIA E-MAIL AND US MAIL

May 10, 2019

Mr. Ken Noyes/Mr. Colin Cardin, Chief Operator  
Winnepesaukee River Basin Program  
P.O.Box 68  
Franklin, NH 03235  
E-mail: [kenneth.noyes@des.nh.gov](mailto:kenneth.noyes@des.nh.gov), [colin.cardin@des.nh.gov](mailto:colin.cardin@des.nh.gov)

Re: Efforts to Respond to Emerging Contaminants such as Per- and Polyfluoroalkyl Substances (PFAS) and Providing Quality Sludge/Biosolids for SQC9706.

Dear, Mr. Ken Noyes/Mr. Colin Cardin

As you may have seen in the news recently there is concern over Emerging Contaminants such as Per- and Polyfluoroalkyl Substances (PFASs) in groundwater, as well as in sludge/biosolids. As part of this, the New Hampshire Department of Environmental Services (NHDES) Drinking Water and Groundwater Bureau (DWGB) will soon adopt rules authorized by legislation enacted in the 2018 legislative session, to establish maximum contaminant levels (MCLs) for four PFAS compounds:

- perfluorooctanoic acid (PFOA),
- perfluorooctane sulfonic acid (PFOS),
- perfluorononanoic acid (PFNA), and
- perfluorohexane sulfonic acid (PFHxS).

To prepare for the regulation of these compounds, NHDES Residuals Management Section (RMS) has investigated the potential impact of these compounds on the NH biosolids program. This letter is sent to explain what NHDES has discovered during our recent investigations, as well as the actions we are taking to enhance the quality of beneficially-used biosolids in NH. Attached please find an updated SQC with additional terms and conditions requiring testing of your certified sludge/biosolids for PFAS compounds annually, and include a narrative of Pollution Prevention and Pretreatment efforts related to PFAS in the SQC9706 Annual Report. Also attached is a new *Sludge Quality Certification (SQC) Fact Sheet* and a Training Announcement from North East Biosolids and Residuals Association (NEBRA).

In the past two years, NHDES has investigated the presence of PFAS compounds in sludge and biosolids. In our research we have detected the presence of all four of the above-listed PFAS compounds in the majority of sludge samples collected by NHDES. All results were well below NHDES's established direct contact limit of 500 parts per billion (ppb) for PFOS and PFOA, and at or below national average concentrations.

NHDES Drinking Water and Groundwater Bureau (DWGB) has also started testing drinking water wells in the vicinity of fields with the historic use of biosolids in agricultural practices, as well as near sludge/biosolids facilities. These results appear to indicate that PFAS do transfer (leach) from biosolids into the soil then into the groundwater; however, the majority of drinking water samples taken near



## SLUDGE QUALITY CERTIFICATION

as authorized by the NH Department of Environmental Services, Water Division (NHDES) pursuant to RSA 485-A and Part Env-Wq 809 of the New Hampshire Sludge Management Rules

I. **GENERATOR IDENTIFICATION:**

**Generator Name/Address:** Winnepesaukee River Basin Program Wastewater Treatment Plant  
P.O. Box 68, Franklin, NH 03235-0068  
**Sludge Quality Certification No.:** SQC-9706  
**Facility Location:** 528 River Street, Franklin, NH 03235-0068  
**Facility Operator Name/Title:** Kenneth W. Noyes, Chief Operator  
**Facility Type/Activities:** Publicly Owned Treatment Works

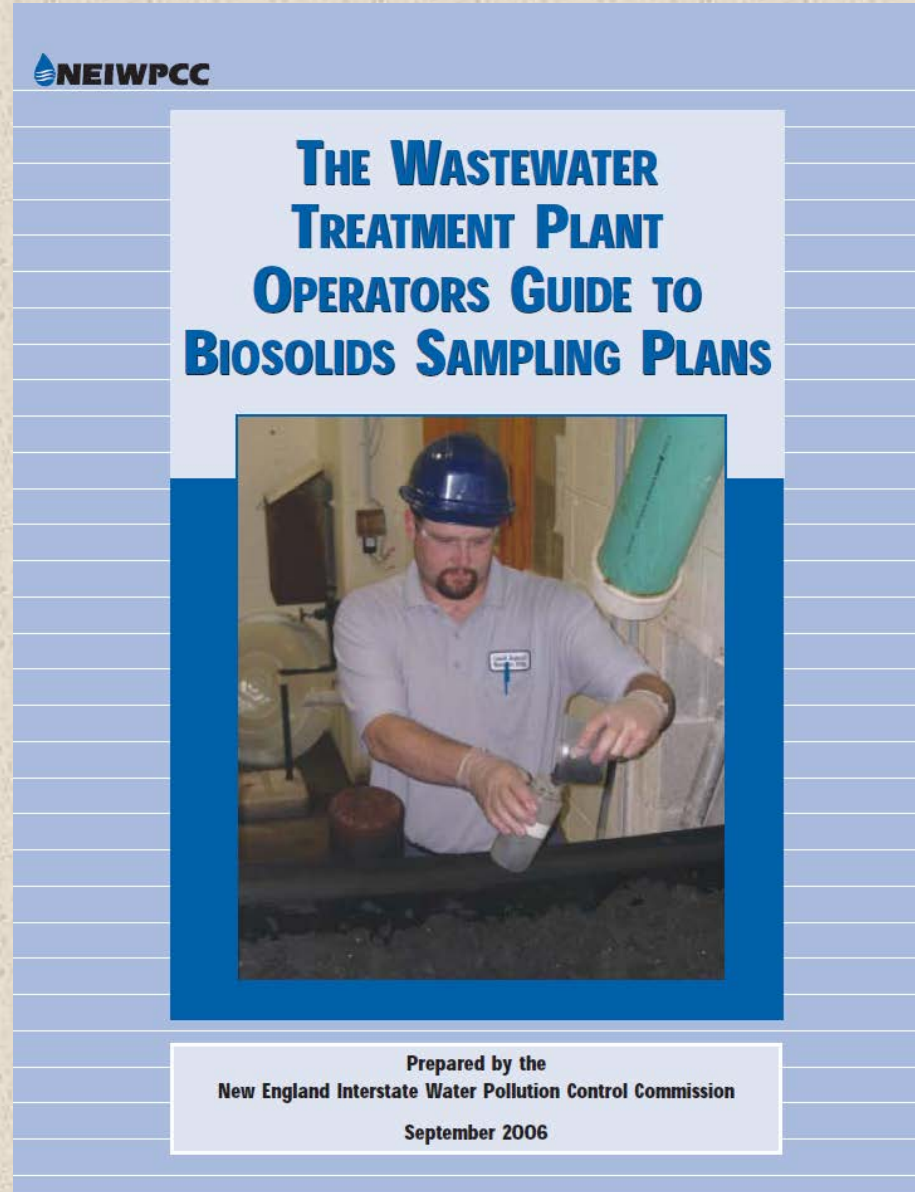
II. **FILE REFERENCE/RECORD OF APPLICATION:**

**Original Certificate Issued:** June 14, 2000  
**Date(s) Application Received:** Received application on November 06, 2015  
Received additional information on January 26 & 29, 2016





# NEIWPCC / NEBRA PFAS Sampling Guidance

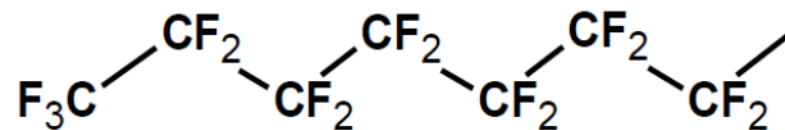




*NEBRA Guidance:*

## **Sampling and Analysis of PFAS in Biosolids and Associated Media** v. 2.0

June 2017 (v. 1.0), updated January 2018 (v. 2.0).



### **Acknowledgements**

This guidance was written and produced by Michael Rainey, M.S. (Northwood, NH) with review and editing by members of the NEBRA PFAS Advisory Group and staff. Special thanks to New England Interstate Water Pollution Control Commission (Lowell, MA) for reliance on their biosolids sampling guide and to Professor Linda S. Lee, Ph.D., Purdue University (West Lafayette, IN).

Category	Prohibited Items	Allowed Items
Pumps and Tubing	Teflon® and other fluoropolymer containing materials, pipe thread seal tape	High-density polyethylene (HDPE), low density polyethylene (LDPE), or silicone tubing, peristaltic pump or stainless steel submersible pump
Decontamination	Decon 90	Alconox® or Liquinox®, potable water followed by deionized rinse.
Sample Storage and Preservation	LDPE or glass bottles, PTFE-or Teflon®-lined caps, chemical ice packs, aluminum foil	Laboratory-provided sample container <i>preferred</i> ; or, HDPE or polypropylene bottles, regular ice sealed in plastic (polyethylene) bags to prevent melt water contaminating samples, thin HDPE sheeting
Field Documentation	Waterproof/treated paper or field books, plastic clipboards, non-Sharpie® markers, Post-It® and other adhesive paper products	Plain Paper, <i>metal</i> clipboard, Sharpies® (allowable per EPA, but other markers are not), pens
Clothing	New or unwashed clothing, clothing or boots made of or with Gore-Tex™ or other synthetic water resistant and/or stain resistant materials, coated Tyvek® material, anything washed with fabric softeners.	Well-laundered synthetic or 100% cotton material, previously laundered clothing (preferably previously washed greater than six times) <i>without</i> the use of fabric softeners . Steel-toed or other boots made with polyurethane and/or polyvinyl chloride (PVC). <i>Uncoated</i> Tyvek.
Personal Care Products (for day of sample collection)	Cosmetics, moisturizers, hand cream, some sunscreens, insect repellants, and other related products, dental floss and plaque removers	Suncscreens: Alba Organics Natural Yes to Cucumbers Aubrey Organics Jason Natural Sun Block Kiss My Face Insect Repellents: Jason Natural Quit Bugging Me Repel Lemon Eucalyptus Herbal Armor California Baby Natural Bug Spray BabyGanics Sunscreen and Insect Repellents: Avon Skin So Soft Bug Guard-SPF 30
Food and Beverage	Pre-packaged food, fast food wrappers or containers, aluminum foil, non-stick cookware & containers	Bottled water or hydration drinks.



## EQUIPMENT CHECKLIST – Biosolids / Residuals / Solids

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1

1) Sample handling and collection

- a. Nitrile gloves
- b. Stainless steel bucket
- c. 500 mL Polypropylene or HDPE container
- d. Stainless steel trowel

2) Transporting and preservation

- a. Sample containers 15 ml graduated polypropylene tubes
- b. Sample cooler with ice

3) Sample ID and Documentation

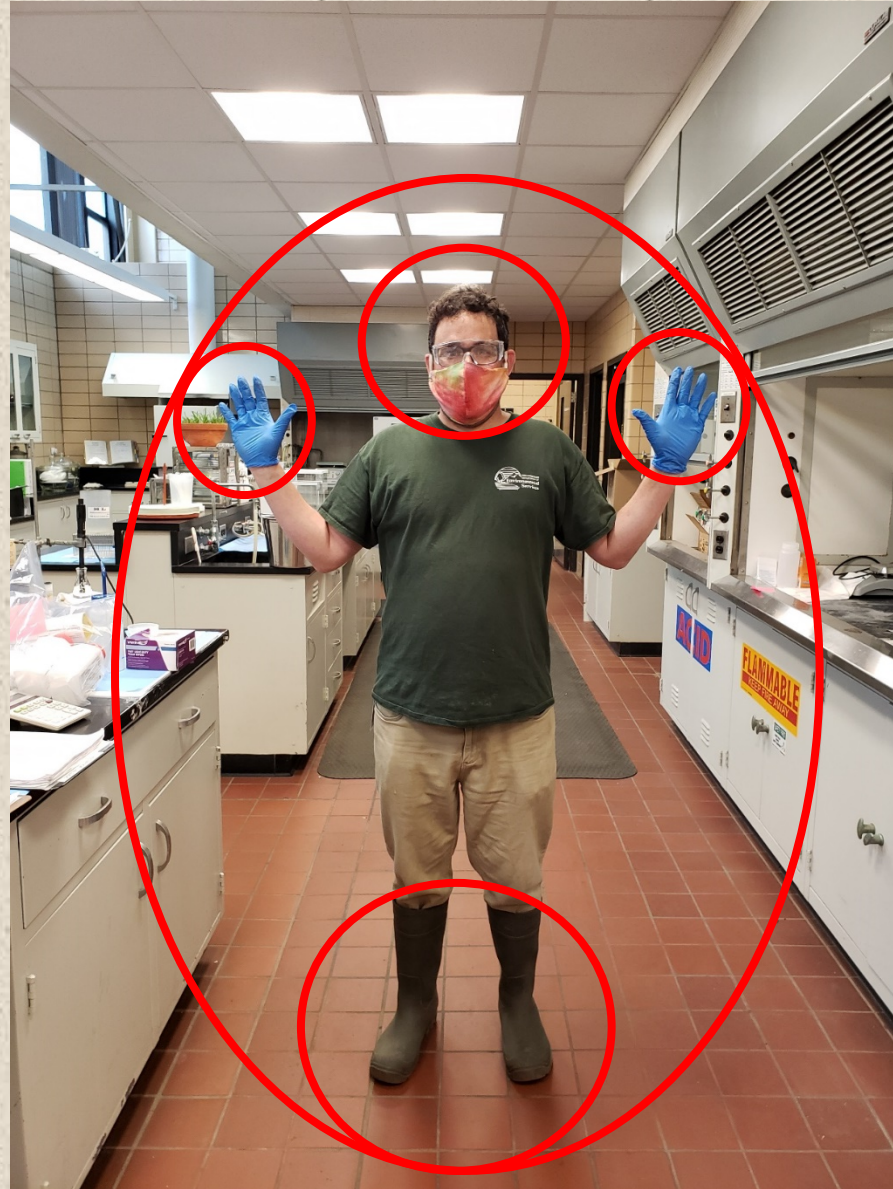
- a. Markers and pens
- b. Sample container labels
- c. Custody seals
- d. Chain of custody/sample submittal form
- e. Field notebook/ sample log/field data sheet

4) Cleaning equipment

- a. Disposable towels
- b. Soap
- c. Scrub brush
- d. Tap water
- e. Deionized water
- f. Methanol
- g. Plastic wrap

5) Ensure that all equipment, supplies, and other materials assembled for sampling, including clothing worn by sampling staff, will not contaminate samples with PFAS extraneous to the residuals being sampled. See **Appendix D-4** for materials that may contain PFAS and that should be avoided.

# PPE and Sampling clothing







# Sample equipment cleaning & sampling

- Rinse equipment with warm tap water to remove most solids.
- Using a brush and PFAS free lab detergent to scrub the equipment to remove all residues
- After scrubbing, rinse the equipment three times with tap water (make sure all detergent is removed).
- The tap water rinse should be followed by rinsing three times with PFAS free deionized water.
- To store, buckets, beakers and other containers can be inverted in a clean, dry location.
- Just prior to sampling, rinse the sample equipment three times in PFAS free deionized water. Take equipment blank rinsate samples to check if your cleaning process is preventing cross contamination.





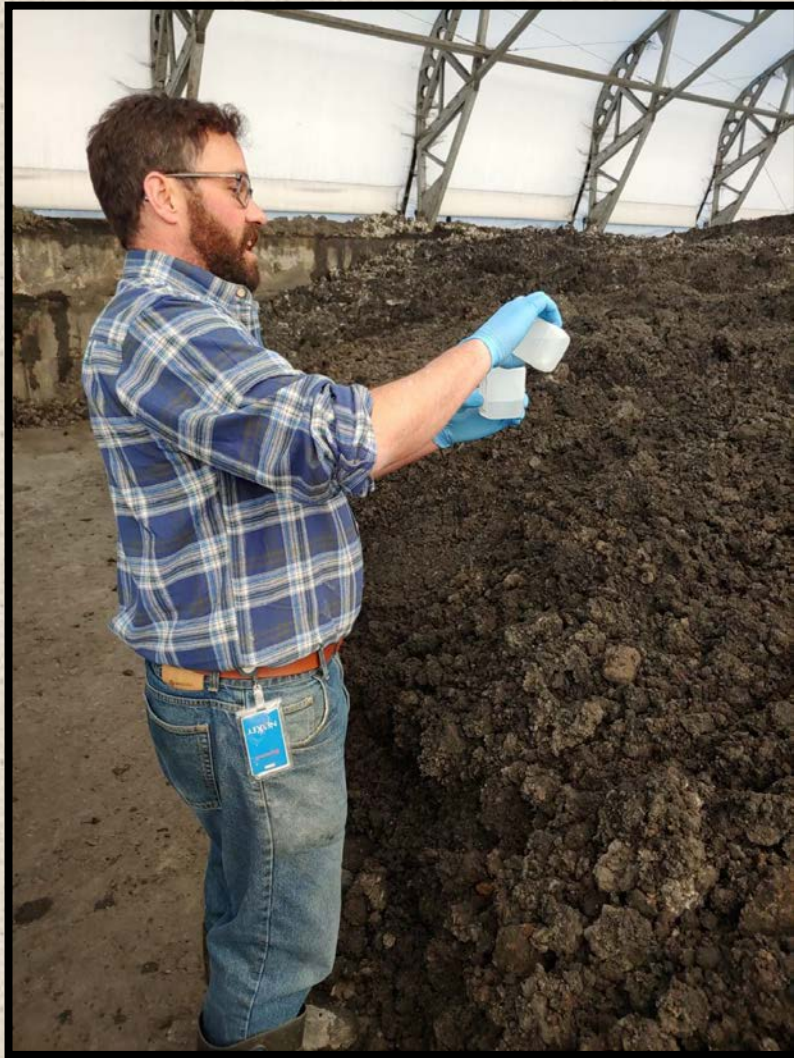




# Equipment and Field Blanks – Sample Quality Checks

- ▶ 11/21/2019: Equipment blank sample with cleaning yielded < 2.36 ng/l for 24 PFAS compounds (biosolids samples: 1<sup>st</sup> sample detected 14 PFAS compounds, 2<sup>nd</sup> biosolids sample detected 6 PFAS compounds)
- ▶ 11/21/2019: Field Blank inside biosolids storage building yielded < 1.87 ng/l for all 24 compounds taken directly in front of sampler and stockpile
- ▶ 06/01/2020: Sludge sample taken in the AM was >50 ng/g (PFOA, PFOS, PFHxS, PFNA)(10 PFAS compounds detected), sample equipment cleaned in permittee's lab then transported to second sample location and triple rinsed with PFAS free DI water, SPF sample taken in PM was <1.11 ng/g for all 24 compounds







Sample Identification	Type (grab or composite)	Sample Time	Analyses
SQC 20001 B-1 Manufactured Top Soil	Composite	11:50 AM	PFAS
Equipment Blank	Grab	12:10 PM	PFAS
SQC13002 Biosolids	Composite	12:25 PM	PFAS
Field Blank	Grab	12:30 PM	PFAS





















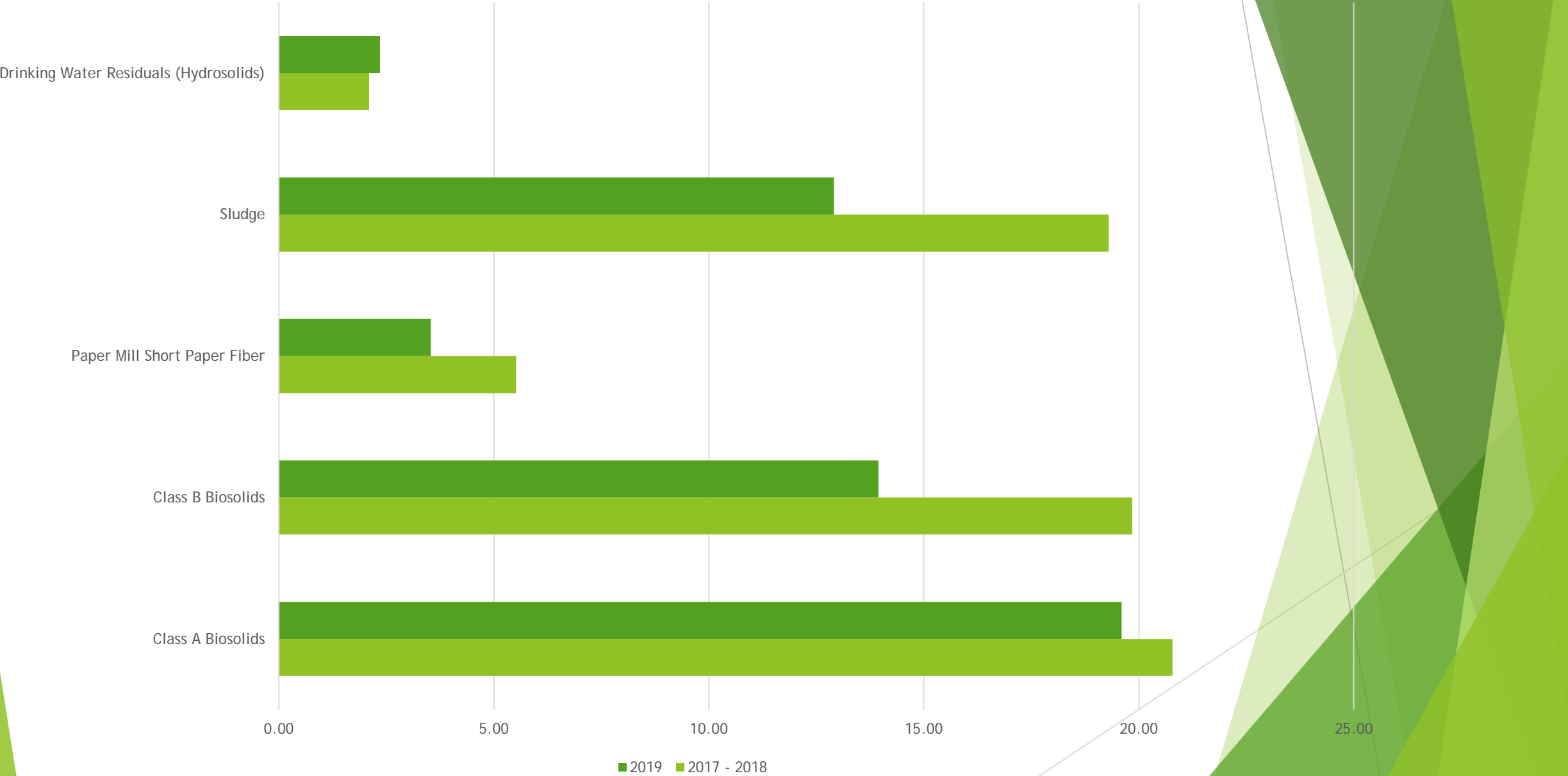








NH SQC 2017 - 2019 PFAS Residuals Investigation (ng/g) (sum of PFOA, PFOS, PFHxS, & PFNA)



Samples analyzed for PFAS were analyzed using isotope dilution. Some compounds from sample reports' QA/QC report were flagged for being outside of percent recovery acceptable criteria.



# Questions or Comments?

