

Trace Analytical Laboratories, Inc.
2241 Black Creek Road
Muskegon, MI 49444-2673



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May 21, 2021

Mr. Tom Rodgers
Benton Harbor- St. Joseph WWTP
269 Anchors Way
St. Joseph, MI 49085

Phone: (269) 363-1070

RE: Trace ID: 21D1049

Dear Mr. Rodgers:

Enclosed are your analytical results associated with your project for Biosolids. The results of this report relate only to the samples listed in the body of this report.

The results were obtained from Merit Laboratories, Inc

Thank you for working with Trace. If you have questions concerning this report, please contact me at 231.773.5998 or by email at tbrewer@trace-labs.com.

Sincerely,

A handwritten signature in black ink that reads "Timothy W. Brewer". The signature is fluid and cursive, with the first name being the most prominent.

Tim Brewer
Project Manager

Enclosures



NJDEP Accreditation No. MI008

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Analytical Laboratory Report

Report ID: S23703.01(01)
Generated on 05/20/2021

Report to

Attention: Tim Brewer
Trace Analytical Laboratories
2241 Black Creek Rd.
Muskegon, MI 49444

Phone: O: 231-773-5998 x238 FAX:
Email: TBrewer@trace-labs.com

Additional Contacts: Jon Mink

Report produced by

Merit Laboratories, Inc.
2680 East Lansing Drive
East Lansing, MI 48823

Phone: (517) 332-0167 FAX: (517) 332-6333

Contacts for report questions:
John Lavery (johnlavery@meritlabs.com)
Barbara Ball (bball@meritlabs.com)

Report Summary

Lab Sample ID(s): S23703.01
Project: 21D1049
Collected Date(s): 04/28/2021
Submitted Date/Time: 04/30/2021 10:20
Sampled by: Unknown
P.O. #: 21D1049

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Maya Murshak
Technical Director



Analytical Laboratory Report

General Report Notes

Analytical results relate only to the samples tested, in the condition received by the laboratory.

Methods may be modified for improved performance.

Results reported on a dry weight basis where applicable.

'Not detected' indicates that parameter was not found at a level equal to or greater than the reporting limit (RL).

40 CFR Part 136 Table II Required Containers, Preservation Techniques and Holding Times for the Clean Water Act specify that samples for acrolein and acrylonitrile need to be preserved at a pH in the range of 4 to 5 or if not preserved, analyzed within 3 days of sampling.

QA/QC corresponding to this analytical report is a separate document with the same Merit ID reference and is available upon request.

Full accreditation certificates are available upon request. Starred (*) analytes are not NELAP accredited.

Samples are held by the lab for 30 days from the final report date unless a written request to hold longer is provided by the client.

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Limits for drinking water samples, are listed as the MCL Limits (Maximum Contaminant Level Concentrations)

PFAS requirement: Section 9.3.8 of U.S. EPA Method 537.1 states "If the method analyte(s) found in the Field Sample is present in the FRB at a concentration greater than 1/3 the MRL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed."

Samples submitted without an accompanying FRB may not be acceptable for compliance purposes.

Report Narrative

There is no additional narrative for this analytical report



Analytical Laboratory Report

Laboratory Certifications

Authority	Certification ID
Michigan DEQ	#9956
DOD ELAP/ISO 17025	#69699
WBENC	#2005110032
Ohio VAP	#CL0002
Indiana DOH	#C-MI-07
New York NELAC	#11814
North Carolina DENR	#680
North Carolina DOH	#26702
Alaska CSLAP	#17-001
Pennsylvania DEP	#68-05884

Qualifier Descriptions

Qualifier	Description
!	Result is outside of stated limit criteria
B	Compound also found in associated method blank
E	Concentration exceeds calibration range
F	Analysis run outside of holding time
G	Estimated result due to extraction run outside of holding time
H	Sample submitted and run outside of holding time
I	Matrix interference with internal standard
J	Estimated value less than reporting limit, but greater than MDL
L	Elevated reporting limit due to low sample amount
M	Result reported to MDL not RDL
O	Analysis performed by outside laboratory. See attached report.
R	Preliminary result
S	Surrogate recovery outside of control limits
T	No correction for total solids
X	Elevated reporting limit due to matrix interference
Y	Elevated reporting limit due to high target concentration
b	Value detected less than reporting limit, but greater than MDL
e	Reported value estimated due to interference
j	Analyte also found in associated method blank
p	Benzo(b)Fluoranthene and Benzo(k)Fluoranthene integrated as one peak.
x	Preserved from bulk sample

Glossary of Abbreviations

Abbreviation	Description
RL/RDL	Reporting Limit
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
SW	EPA SW 846 (Soil and Wastewater) Methods
E	EPA Methods
SM	Standard Methods
LN	Linear
BR	Branched

Method Summary

Method	Version
ASTM D7968-17M	ASTM Method D7968 - 17 Modified (Isotopic Dilution)
SM2540B	Standard Method 2540 B 2011

Parameter Summary

Parameter	Synonym	Cas #
PFBA	Perfluorobutanoic Acid	375-22-4
PFPeA	Perfluoropentanoic Acid	2706-90-3
4:2 FTSA	4:2 Fluorotelomer Sulfonic Acid	757124-72-4
PFHxA	Perfluorohexanoic Acid	307-24-4
PFBS	Perfluorobutane sulfonic Acid	375-73-5
PFHpA	Perfluoroheptanoic Acid	375-85-9
PFPeS	Perfluoropentane Sulfonic Acid	2706-91-4
6:2 FTSA	6:2 Fluorotelomer Sulfonic Acid	27619-97-2
PFOA	Perfluorooctanoic Acid	335-67-1
PFHxS	Perfluorohexane Sulfonic Acid	355-46-4
PFHxS-LN	Perfluorohexane Sulfonic Acid - LN	355-46-4-LN
PFHxS-BR	Perfluorohexane Sulfonic Acid - BR	355-46-4-BR
PFNA	Perfluorononanoic Acid	375-95-1
8:2 FTSA	8:2 Fluorotelomer Sulfonic Acid	39108-34-4
PFHpS	Perfluoroheptane Sulfonic Acid	375-92-8
PFDA	Perfluorodecanoic Acid	335-76-2
N-MeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9
EtFOSAA	N-Ethyl Perfluorooctane Sulfonamidoacetic Acid	2991-50-6
PFOS	Perfluorooctane Sulfonic Acid	1763-23-1
PFOS-LN	Perfluorooctane Sulfonic Acid - LN	1763-23-1-LN
PFOS-BR	Perfluorooctane Sulfonic Acid - BR	1763-23-1-BR
PFUnDA	Perfluoroundecanoic Acid	2058-94-8
PFNS	Perfluorononane Sulfonic Acid	68259-12-1
PFDoDA	Perfluorododecanoic Acid	307-55-1
PFDS	Perfluorodecane Sulfonic Acid	335-77-3
PFTTrDA	Perfluorotridecanoic Acid	72629-94-8
FOSA	Perfluorooctane Sulfonamide	754-91-6
PFTeDA	Perfluorotetradecanoic Acid	376-06-7
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone1-sulfonic acid	756426-58-1
ADONA	4,8-dioxa-3H-perfluorononanoic acid	919005-14-4
HFPO-DA	Hexafluoropropylene oxide dimer	13252-13-6



Analytical Laboratory Report

Sample Summary (1 samples)

Sample ID	Sample Tag	Matrix	Collected Date/Time
S23703.01	BHSJ Biosolids 21D1049-01	Sludge	04/28/21 13:00



Analytical Laboratory Report

Lab Sample ID: S23703.01

Sample Tag: BHSJ Biosolids 21D1049-01

Collected Date/Time: 04/28/2021 13:00

Matrix: Sludge

COC Reference:

Sample Containers

#	Type	Preservative(s)	Refrigerated?	Arrival Temp. (C)	Thermometer #
1	15ml Centrifuge Tube	None	Yes	3.8	IR
1	250ml Plastic	None	Yes	3.8	IR

Extraction / Prep.

Parameter	Result	Method	Run Date	Analyst	Flags
Initial wt. (g) / Final wt. (g) / Volume (ml)*	8.44/6.94/10	ASTM D7968-17M	05/17/21 14:45	KCV	

Inorganics

Method: SM2540B, Run Date: 05/03/21 12:00, Analyst: ELR

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
Total Solids*	1.9	1		%	1		

Organics

28 PFAs, Method: ASTM D7968-17M, Run Date: 05/18/21 04:28, Analyst: KCV

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
PFBA*	Not detected	7		ug/kg	351	375-22-4	
PFPeA*	Not detected	3.5		ug/kg	351	2706-90-3	
4:2 FTSA*	Not detected	3.5		ug/kg	351	757124-72-4	I
PFHxA*	Not detected	3.5		ug/kg	351	307-24-4	
PFBS*	Not detected	3.5		ug/kg	351	375-73-5	
PFHpA*	Not detected	3.5		ug/kg	351	375-85-9	
PFPeS*	Not detected	3.5		ug/kg	351	2706-91-4	
6:2 FTSA*	Not detected	3.5		ug/kg	351	27619-97-2	I
PFOA*	Not detected	3.5		ug/kg	351	335-67-1	
PFHxS*	Not detected	3.5		ug/kg	351	355-46-4	
PFHxS-LN*	Not detected	3.5		ug/kg	351	355-46-4-LN	
PFHxS-BR*	Not detected	3.5		ug/kg	351	355-46-4-BR	
PFNA*	Not detected	3.5		ug/kg	351	375-95-1	
8:2 FTSA*	Not detected	3.5		ug/kg	351	39108-34-4	I
PFHpS*	Not detected	3.5		ug/kg	351	375-92-8	
PFDA*	Not detected	3.5		ug/kg	351	335-76-2	
N-MeFOSAA*	9.9	3.5		ug/kg	351	2355-31-9	I
EtFOSAA*	6.2	3.5		ug/kg	351	2991-50-6	I
PFOS*	11	3.5		ug/kg	351	1763-23-1	
PFOS-LN*	8.6	3.5		ug/kg	351	1763-23-1-LN	
PFOS-BR*	Not detected	3.5		ug/kg	351	1763-23-1-BR	
PFUnDA*	Not detected	3.5		ug/kg	351	2058-94-8	
PFNS*	Not detected	3.5		ug/kg	351	68259-12-1	
PFDoDA*	Not detected	3.5		ug/kg	351	307-55-1	
PFDS*	Not detected	3.5		ug/kg	351	335-77-3	
PFTTrDA*	Not detected	3.5		ug/kg	351	72629-94-8	
FOSA*	Not detected	3.5		ug/kg	351	754-91-6	
PFTeDA*	Not detected	3.5		ug/kg	351	376-06-7	
11Cl-PF3OUdS*	Not detected	3.5		ug/kg	351	763051-92-9	
9Cl-PF3ONS*	Not detected	3.5		ug/kg	351	756426-58-1	

I-Matrix interference with internal standard



Analytical Laboratory Report

Lab Sample ID: S23703.01 (continued)
Sample Tag: BHSJ Biosolids 21D1049-01

28 PFAs, Method: ASTM D7968-17M, Run Date: 05/18/21 04:28, Analyst: KCV (continued)

Parameter	Result	RL	MDL	Units	Dilution	CAS#	Flags
ADONA*	Not detected	3.5		ug/kg	351	919005-14-4	
HFPO-DA*	Not detected	3.5		ug/kg	351	13252-13-6	

Merit Laboratories Login Checklist

Lab Set ID:S23703

Client:TRACE (Trace Analytical Laboratories)

Project: 21D1049

Submitted:04/30/2021 10:20 Login User: REJ

Attention: Tim Brewer

Address: Trace Analytical Laboratories
2241 Black Creek Rd.
Muskegon, MI 49444

Phone: O: 231-773-5998 FAX:

Email: TBrewer@trace-labs.com

Selection	Description	Note
Sample Receiving		
01.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples are received at 4C +/- 2C Thermometer # IR 3.5
02.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Received on ice/ cooling process begun
03.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples shipped UPS
04.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples left in 24 hr. drop box
05.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Are there custody seals/tape or is the drop box locked
Chain of Custody		
06.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC adequately filled out
07.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	COC signed and relinquished to the lab
08.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sample tag on bottles match COC
09.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Subcontracting needed? Subcontracted to:
Preservation		
10.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Do sample have correct chemical preservation
11.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Completed pH checks on preserved samples? (no VOAs)
12.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Did any samples need to be preserved in the lab?
Bottle Conditions		
13.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	All bottles intact
14.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Appropriate analytical bottles are used
15.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Merit bottles used
16.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Sufficient sample volume received
17.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Samples require laboratory filtration
18.	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Samples submitted within holding time
19.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Do water VOC or TOX bottles contain headspace

Corrective action for all exceptions is to call the client and to notify the project manager.

Client Review By: _____ Date: _____

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Muskegon, MI 49444-2673



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SUBCONTRACT ORDER

21D1049

SENDING LABORATORY:

Trace Analytical Laboratories, Inc.
2241 Black Creek Road
Muskegon, MI 49444
Phone: 231.773.5998

RECEIVING LABORATORY:

Merit Laboratories, Inc
2680 East Lansing Dr.
East Lansing, MI 48823
Phone :(517) 332-0167

Project Manager: Tim Brewer

PO # 21D1049

23703.01

Sample ID: BHSJ Biosolids 21D1049-01

Matrix: Sludge

Sampled: 04/28/21 13:00

TAT: Standard

Analysis Needed:

PFAS- Biosolids- EGLE List

(Trace) MTR

Released By

Date

4/29/21

WBS

4/28/21

Received By

WBS

Date

4/30/21 1020

Released By

Date

3.5

Trace ID No. 21D1040

Logged By:	NC
Checked By:	WJL
Soil Volatiles Preserved (circle if applicable):	
MeOH	Low Level
	Lab
Sampling Time:	

[illegible]

Report ID: 21D1049 TRACE Farmed Out FINAL 05 21 21 0621

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SAMPLE LOG IN CHECKLIST

Trace ID #: <u>21D1049</u>	Date: <u>4/29/21</u>	Package Description: <u>Cooler</u>	Temperature: <u>-0.4</u>
Client Name: <u>BHSJ WWTP</u>	Time: <u>12:43</u>	Logged in by: <u>JK</u>	

Cooler Receipt

Cooler/samples delivered by:	Trace courier <input checked="" type="checkbox"/>	Hand delivered <input type="checkbox"/>	Commercial courier <input type="checkbox"/>	UPS <input type="checkbox"/>	FED EX <input type="checkbox"/>	US Mail <input type="checkbox"/>
			Name of delivery person: <u>Grant Hilleary</u>			
Tracking Number:			<input checked="" type="checkbox"/> Not Applicable			
Tracking #:						
COC Seals present and intact on cooler?			<input checked="" type="checkbox"/> Not Applicable <input type="checkbox"/> No <input type="checkbox"/> Yes			
Custody seals signed by Client?			<input type="checkbox"/> No <input type="checkbox"/> Yes Client custody seal # (if applicable): _____			

Coolant and Temperature

Type of Coolant Used		Cooler Temperature	
Slurry w/ crushed, cubed, or chip ice?	<input type="checkbox"/>	Correction Factors:	• Digital Stick Thermometer CF = -0.4°C (20B12743)
Multiple bags of ice around samples?	<input checked="" type="checkbox"/>		• IR Thermometer CF = -0.5°C (IR #8)
Ice Packs/ Blue Ice:	<input type="checkbox"/>	Representative Sample Temperature:	<u>1.0</u> °C (check one below)
No Coolant Present:	<input type="checkbox"/>	<input checked="" type="checkbox"/> Temp Blank (Stick Thermometer)	
Ice still present upon receipt (circle one):	<u>Yes</u> No N/A	<input type="checkbox"/> Client Sample (IR Thermometer)	
		Melt Water:	<u>none</u> °C (Use Digital Stick Thermometer)

General

	Yes	No	NA	Comments
All bottles arrived unbroken with labels in good condition?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Each sample point is in a sealed plastic bag?	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<u>NC 4/29/21</u>
Labels filled out completely?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
All bottle labels agree with Chain of Custody (COC)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Sufficient sample to run tests requested?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
*pH checked - samples at correct pH and labeled as such?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Correct chemical preservative added to samples?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Air bubbles absent from VOAs?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
COC filled out properly and signed by client?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
COC signed in by TRACE sample custodian?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was project manager called and samples discussed?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

Notes:

*EMD pH Test Strips Used:

☐ pH 0-2.5 Lot: HC029115 ☐ pH 11.0-13.0 Lot: HC729101
☐ Other: _____

Form 70-A.37
Effective 4/20/21

TRACE Analytical Laboratories, Inc.

CERTIFICATE OF ANALYSIS

This report shall not be reproduced, except in full, without the written consent of Trace Analytical Laboratories, Inc.

Report ID: 21D1049 TRACE_Farmed_Out FINAL 05 21 21 0621