

State of Washington Department of Ecology
Northwest Regional Office
WATER COMPLIANCE INSPECTION REPORT

substitute for OMB No. 2040-0057 and EPA form 3560-3 (Rev. 9-94) (last file update 12-95.)

Section A: National Data System Coding (i.e., PCS)

Transaction Code 1 N 2 5	NPDES # WA0501489	yr/mo/day 12 14/06/12	Inspection Type 18 S	Inspector 19 S	Fac Type 20 1
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Remarks

Pending Application

Inspection work days 67 1.0 69	Facility Self-Monitoring Evaluation Rating 70 4	BI 71 N	QA 72 N	-----Reserved-----			
		73	74	75	80		

Section B: Facility Data

Name and Location of Facility Inspected (For industrial users discharging to POTW, also include POTW name and NPDES permit number) Hughes Farms 1325 Farm to Market Road Mount Vernon, WA 98273	Entry Time/Date 11:30 am 06/12/14	Permit Effective Date
	Exit Time / Date 1:00 pm 06/12/14	Permit Expiration Date

Name(s) of On-Site Representative(s)/Title(s)/Phone and Fax Number(s) Jose Velazquez Plant Manager 360-424-3772	Other Facility Data
Name, Address of Responsible Official/Title/Phone and Fax Number. David Hughes Owner	
Phone Number: (360) 424-3772 Fax: Contacted? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	

Section C: Areas Evaluated During Inspection (Check only those areas evaluated)

<input type="checkbox"/> Permit	<input type="checkbox"/> Flow Measurement	<input type="checkbox"/> Operations & Maint.	<input type="checkbox"/> CSO/SSO (Sewer Overflow)
<input type="checkbox"/> Records/Reports	<input type="checkbox"/> Self-Monitoring Program	<input type="checkbox"/> Sludge Handling/Disposal	<input type="checkbox"/> Pollution Prevention
<input type="checkbox"/> Facility Site Review	<input type="checkbox"/> Compliance Schedules	<input type="checkbox"/> Pretreatment	<input type="checkbox"/> Multimedia
<input checked="" type="checkbox"/> Effluent/Receiving water	<input type="checkbox"/> Laboratory	<input type="checkbox"/> Storm Water	<input type="checkbox"/> other

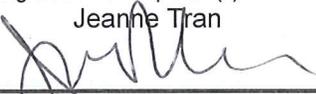
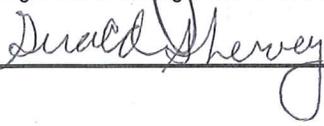
Section D: Summary of Findings/Comments

INTRODUCTION

This was a class II inspection involving collecting water samples to investigate the cause of the discoloration and odor of the water in the field ditch and the settling pond. The sample was collected in the water column at a depth of approximately 1 foot. Samples were analyzed for turbidity, BOD₅, sulfide, sulfite, sulfate, sulfur, ammonia, nitrate/nitrite, TPN, fecal coliforms, manganese (total and dissolved metal). Attached are the analytical results.

Background information: The settling pond is shared by Hughes Farms, Chemtrade Sulex, and other nearby facilities. The majority of the flow comes from Hughes Farms' potato washing operation. Occasionally, other facilities discharge stormwater runoff into this pond. Hughes Farms has been pumping water from the settling pond to the farm field for irrigation.

In summary, the analytical results indicate that the BOD₅ concentrations in the field ditch and in the settling pond were high compared to the municipal technology-based standards for surface water discharge (30 mg/L average monthly, and 45 mg/L average weekly). The measured DO concentration in the settling pond was very low (less than 2 mg/L) compared to the surface water standard (8 mg/L). The reason for the discoloration of the water is unknown. Nevertheless, a common cause of black discoloration of water is due to the formation of sulfides of multivalent metals (e.g. manganese sulfide, iron sulfide, magnesium sulfide, etc). However, the sulfide concentration in the sample was less than the sulfite and sulfate concentrations. Furthermore, Chemtrade Sulex has not been discharging to the settling pond since 2012 and the pond is dredged every year at the end of August or early September by Hughes Farms. In addition, I did not detect a rotten egg odor when I was present on-site on June 9, 2014. My hypothesis is the combination of the nutrients from the decomposition of potato pieces, and the starch and sugars from potatoes coupled with the uncharacteristically warm temperature lead to a predominantly anaerobic condition in the lower portion of the water column, and in the benthos of the pond, resulting in the formation of metallic sulfides which could be the cause of the black discoloration of the water.

Name(s) and Signatures of Inspector(s) Jeanne Tran 	Agency/Office/Telephone WA State Dept. of Ecology/NWRO/(425)649-7078 3190 160th SE Ave, Bellevue, WA 98008-5452	Date 09/26/14
Signature of Management Q A Reviewer 	Agency/Office/Phone and Fax Numbers WA Dept. of Ecology/NWRO/(425)649-7000 fax (425)649-7098	Date 10/13/2014

UNANNOUNCED Inspection

INSTRUCTIONS

Section A: National Data System Coding (i.e., PCS)

Column 1: Transaction Code. Use N, C, or D for New Change or Delete. All inspections will be new unless there is an error in the data entered.

Columns 3-11: NPDES Permit No. Enter the facility's NPDES permit number. (Use the Remarks columns to record State permit number, if necessary.)

Columns 12-17: Inspection Date. Insert the date entry was made into the facility. Use the year/month/day format (e.g., 94/06/30 = June 30, 1994).

Column 18: Inspection Type. Use one of the codes listed below to describe the type of inspection:

A Performance Audit	L Enforcement Case Support	2 IU Sampling Inspection
B Compliance Biomonitoring	M Multimedia	3 IU Non-Sampling Inspection
C Compliance Evaluation (non-sampling)	P Pretreatment Compliance Inspection	4 IU Toxics Inspection
D Diagnostic	R Reconnaissance	5 IU Sampling Inspection with Pretreatment
E Corps of Engineers Inspection	S Compliance Sampling	6 IU Non-Sampling Inspection with pretreatment
F Pretreatment Follow-up	U IU Inspection with Pretreatment Audit	7 IU Toxics with Pretreatment
G Pretreatment Audit	X Toxics Inspection	
I Industrial User (IU) Inspection	Z Sludge	

Column 19: Inspector Code. Use one of the codes listed below to describe the *lead agency* in the inspection.

C - Contractor or Other Inspectors (Specify in Remarks Columns)	N - NEIC Inspectors
E - Corps of Engineers	R - EPA Regional Inspector
J - Joint EPA/State Inspectors - EPA Lead	S - State Inspector
	T - Joint State/EPA Inspectors - State Lead

Column 20: Facility Type. Use one of the codes below to describe the facility.

- 1 - Municipal. Publicly Owned Treatment Works (POTWs) with 1987 Standard Industrial Code (SIC) 4952.
- 2 - Industrial. Other than municipal, agricultural, and Federal facilities.
- 3 - Agricultural. Facilities classified with 1987 SIC 0111 to 0971.
- 4 - Federal. Facilities identified as Federal by the EPA Regional Office

Columns 21-66: Remarks. These columns are reserved for remarks at the discretion of the Region.

Columns 67-69: Inspection Work Days. Estimate the total work effort (to the nearest 0.1 work day), up to 99.9 days, that were used to complete the inspection and submit a QA reviewed report of findings. This estimate includes the accumulative effort of all participating inspectors; any effort for laboratory analyses, testing, and remote sensing; and the billed payroll time for travel and pre and post inspection preparation. This estimate does not require detailed documentation.

Column 70: Facility Evaluation Rating. Use information gathered during the inspection (regardless of inspection type) to evaluate the quality of the facility self-monitoring program. Grade the program using a scale of 1 to 5 with a score of 5 being used for very reliable self-monitoring programs, 3 being satisfactory, and 1 being used for very unreliable programs.

Column 71: Biomonitoring Information. Enter D for static testing. Enter F for flow through testing. Enter N for no biomonitoring.

Column 72: Quality Assurance Data Inspection. Enter Q if the inspection was conducted as follow-up on quality assurance sample results. Enter N otherwise.

Columns 73-80: These columns are reserved for regionally defined information.

Section B: Facility Data

This section is self-explanatory except for "Other Facility Data," which may include new information not in the permit or PCS (e.g., new outfalls, names of receiving waters, new ownership, and other updates to the record).

Section C: Areas Evaluated During Inspection

Check only those areas evaluated by marking the appropriate box. Use Section D and additional sheets as necessary. Support the findings, as necessary, in a brief narrative report. Use the headings given on the report form (e.g., Permit, Records/Reports) when discussing the areas evaluated during the inspection. The heading marked "Multimedia" may indicate medias such as CAA, RCRA, and TSCA. The heading marked "Other" may indicate activities such as SPCC, BMPs, and concerns that are not covered elsewhere.

Section D: Summary of Findings/Comments

Briefly summarize the inspection findings. This summary should abstract the pertinent inspection findings, not replace the narrative report. Reference a list of attachments, such as completed checklists taken from the NPDES Compliance Inspection Manuals and pretreatment guidance documents, including effluent data when sampling has been done. Use extra sheets as necessary.



Analytical Resources, Incorporated
Analytical Chemists and Consultants

June 20, 2014

Karin Feddersen
Washington State Department of Ecology
7411 Beach Drive East
Port Orchard, WA 98366-8204

RE: Project: Black Water
ARI Job No.: YN65

Dear Karin:

Please find enclosed the original Chain-of-Custody record (COC), sample receipt documentation, and the final data package for samples from the project referenced above. For information regarding sample receipt, refer to the Cooler Receipt Form.

Analytical details are discussed in the Case Narrative.

An electronic copy of this package will remain on file with ARI. Should you have any questions or problems, please feel free to contact me at your convenience.

Sincerely,

ANALYTICAL RESOURCES, INC.

A handwritten signature in black ink, appearing to read "Cheronne Oreiro".

Cheronne Oreiro
Project Manager
(206) 695-6214
cheronneo@arilabs.com
www.arilabs.com

cc: eFile YN65

Enclosures



Cooler Receipt Form

ARI Client WSDOE

Project Name Black Water

COC No(s) _____ (NA)

Delivered by: Fed-Ex UPS Courier (Hand Delivered) Other _____

Assigned ARI Job No: _____

Tracking No: _____ (NA)

Preliminary Examination Phase:

Were intact, properly signed and dated custody seals attached to the outside of to cooler? YES NO

Were custody papers included with the cooler? YES NO

Were custody papers properly filled out (ink, signed, etc) YES NO

Temperature of Cooler(s) (°C) (recommended 2.0-6.0 °C for chemistry)

Time: 1700

238

If cooler temperature is out of compliance fill out form 00070F

Temp Gun ID#: 90877952

Cooler Accepted by AV Date: 6/12/14 Time: 1700

Complete custody forms and attach all shipping documents

Log-In Phase:

Was a temperature blank included in the cooler? YES NO

What kind of packing material was used? Bubble Wrap Wet Ice Gel Packs Baggies Foam Block Paper Other: _____

Was sufficient ice used (if appropriate)? NA YES NO

Were all bottles sealed in individual plastic bags? YES NO

Did all bottles arrive in good condition (unbroken)? YES NO

Were all bottle labels complete and legible? YES NO

Did the number of containers listed on COC match with the number of containers received? YES NO

Did all bottle labels and tags agree with custody papers? YES NO

Were all bottles used correct for the requested analyses? YES NO

Do any of the analyses (bottles) require preservation? (attach preservation sheet, excluding VOCs)... NA YES NO

Were all VOC vials free of air bubbles? NA YES NO

Was sufficient amount of sample sent in each bottle? YES NO

Date VOC Trip Blank was made at ARI NA YES NO

Was Sample Split by ARI: NA YES Date/Time: _____ Equipment: _____ Split by: _____

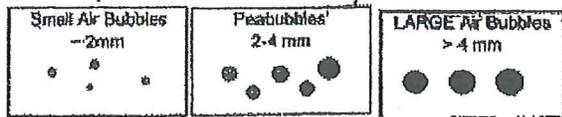
Samples Logged by: AV Date: 6/12/14 Time: 1700

**** Notify Project Manager of discrepancies or concerns ****

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Additional Notes, Discrepancies, & Resolutions:

Field Ditch time on bottles (sulfite) = 1155, No time on (sulfide)
 Settling Pond time on bottle (sulfite) = 1257, (sulfide) = 1255
 By: AV/CO Date: 6/12/13 Per the client, Field Ditch was logged as 1406053-01 +
 Settling Pond was logged as 1406053-02. Only 2 samples rec'd 6/12/14.



- Small → "sm" (< 2 mm)
- Peabubbles → "pb" (2 to < 4 mm)
- Large → "lg" (4 to < 6 mm)
- Headspace → "hs" (> 6 mm)



REQUEST FOR LABORATORY SERVICES

PAGE 1 OF 1

SIC NO. _____

Quote: **Contract# 02413**

LIMS ID: _____

PPR Request ID

Contact Cheronne Oreiro Laboratory Analytical Resources, Inc Address 4611 S. 134th Place, Suite 100 Tukwila, WA 98168-3240 Phone: (206)695-6214 FAX: () -	Project Name Black Water Client and Address: WA STATE DEPT. OF ECOLOGY MANCHESTER LABORATORY 7411 BEACH DRIVE EAST PORT ORCHARD WA 98366-8204	Date 06/12/14 <input type="checkbox"/> Enforcement <input type="checkbox"/> Return to Client <input type="checkbox"/> Dispose Return Cooler
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ITEM NO.	LAB SAMPLE NO	PROJECT NAME AND/OR DESCRIPTION	QUANTITY	UNIT PRICE	TOTAL COST
	1406053 - 01,02,03	Analyze 3 water samples collected 06/12/14 for Sulfite by Method SM 4500-S2.	3	26.00	78.00
	1406053 - 01,02,03	Analyze 3 water samples collected 06/12/14 for Sulfide by Method SM4500-SO3.	3	30.00	90.00
		Deliverables to include a case narrative documenting any sample or analysis anomalies, copies of all raw data and QA/QC summaries.	15%	\$3.78	\$3.78
		Report is due within fifteen (15) business days of the sample receipt.			
		Please send invoice with report.			
			TOTAL		\$171.78

Requested By (Your contact if any question arise): Nancy Rosenbower	Phone No. (360) 871-8827
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CHAIN OF CUSTODY*

Relinquished By:	Received By:	Yr	Mo	Day	Hr	Min

*Signatures on this part of the form pertain to the custody of these samples and not to the cost of the analysis.
 Invoice will be paid after sample analyses have passed a QA/QC review.

YNS5 : 00005



ARI Job No: YN65

PC: Cheronne
VTSR: 06/12/14

Inquiry Number: NONE
Analysis Requested: 06/13/14
Contact: Feddersen, Karin
Client: Washington Department of Ecology
Logged by: AV
Sample Set Used: Yes-481
Validatable Package: No
Deliverables:

Project #: 1406053
Project: Black Water
Sample Site:
SDG No:
Analytical Protocol: In-house

LOGNUM	ARI ID	CLIENT ID	CN	WAD	NH3	COD	FOG	MET	PHEN	PHOS	TKN	NO23	TOC	S2	TPHD	Fe2+	DMET DOC	ADJUSTED TO	LOT NUMBER	AMOUNT ADDED	DATE/BY
14-11372	YN65A	FIELD DITCH	>12	>12	<2	<2	<2	<2	<2	<2	<2	<2	<2	>9	<2	<2		<12	10249C	20L	6/12/14
14-11373	YN65B	SETTLING POND												*				I	6/12/14	I	I

* Sulfide preserved w/ ZnCl2, lab to adjust Ph.

ARD 6/12/14

Checked By A Date 6/12/14



Case Narrative

Client: Washington State Department of Ecology
Project: Black Water
ARI Job No.: YN65

Sample receipt

Two water samples were received on June 12, 2014 under ARI job YN65. The cooler temperature measured by IR thermometer following ARI SOP was 23.8°C. For further details regarding sample receipt, please refer to the enclosed Cooler Receipt Form.

General Chemistry Parameters: Sulfide and Sulfite

The samples and associated laboratory QC were analyzed within the recommended holding time.

The method blanks were clean at the reporting limits. The LCS percent recoveries were within limits.

The matrix spike percent recovery and replicate RPDs were within control limits.

Sample ID Cross Reference Report



ARI Job No: YN65
Client: Washington Department of Ecology
Project Event: 1406053
Project Name: Black Water

Sample ID	ARI Lab ID	ARI LIMS ID	Matrix	Sample Date/Time	VTSR
1. FIELD DITCH	YN65A	14-11372	Water	06/12/14 11:33	06/12/14 17:00
2. SETTLING POND	YN65B	14-11373	Water	06/12/14 12:57	06/12/14 17:00



Data Reporting Qualifiers

Effective 2/14/2011

Inorganic Data

- U Indicates that the target analyte was not detected at the reported concentration
- * Duplicate RPD is not within established control limits
- B Reported value is less than the CRDL but \geq the Reporting Limit
- N Matrix Spike recovery not within established control limits
- NA Not Applicable, analyte not spiked
- H The natural concentration of the spiked element is so much greater than the concentration spiked that an accurate determination of spike recovery is not possible
- L Analyte concentration is ≤ 5 times the Reporting Limit and the replicate control limit defaults to ± 1 RL instead of the normal 20% RPD

Organic Data

- U Indicates that the target analyte was not detected at the reported concentration
- * Flagged value is not within established control limits
- B Analyte detected in an associated Method Blank at a concentration greater than one-half of ARI's Reporting Limit or 5% of the regulatory limit or 5% of the analyte concentration in the sample.
- J Estimated concentration when the value is less than ARI's established reporting limits
- D The spiked compound was not detected due to sample extract dilution
- E Estimated concentration calculated for an analyte response above the valid instrument calibration range. A dilution is required to obtain an accurate quantification of the analyte.
- Q Indicates a detected analyte with an initial or continuing calibration that does not meet established acceptance criteria ($< 20\%$ RSD, $< 20\%$ Drift or minimum RRF).



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- S Indicates an analyte response that has saturated the detector. The calculated concentration is not valid; a dilution is required to obtain valid quantification of the analyte
- NA The flagged analyte was not analyzed for
- NR Spiked compound recovery is not reported due to chromatographic interference
- NS The flagged analyte was not spiked into the sample
- M Estimated value for an analyte detected and confirmed by an analyst but with low spectral match parameters. This flag is used only for GC-MS analyses
- M2 The sample contains PCB congeners that do not match any standard Aroclor pattern. The PCBs are identified and quantified as the Aroclor whose pattern most closely matches that of the sample. The reported value is an estimate.
- N The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification"
- Y The analyte is not detected at or above the reported concentration. The reporting limit is raised due to chromatographic interference. The Y flag is equivalent to the U flag with a raised reporting limit.
- EMPC Estimated Maximum Possible Concentration (EMPC) defined in EPA Statement of Work DLM02.2 as a value "calculated for 2,3,7,8-substituted isomers for which the quantitation and /or confirmation ion(s) has signal to noise in excess of 2.5, but does not meet identification criteria"
(Dioxin/Furan analysis only)
- C The analyte was positively identified on only one of two chromatographic columns. Chromatographic interference prevented a positive identification on the second column
- P The analyte was detected on both chromatographic columns but the quantified values differ by $\geq 40\%$ RPD with no obvious chromatographic interference
- X Analyte signal includes interference from polychlorinated diphenyl ethers.
(Dioxin/Furan analysis only)
- Z Analyte signal includes interference from the sample matrix or perfluorokerosene ions. **(Dioxin/Furan analysis only)**



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Geotechnical Data

- A The total of all fines fractions. This flag is used to report total fines when only sieve analysis is requested and balances total grain size with sample weight.
- F Samples were frozen prior to particle size determination
- SM Sample matrix was not appropriate for the requested analysis. This normally refers to samples contaminated with an organic product that interferes with the sieving process and/or moisture content, porosity and saturation calculations
- SS Sample did not contain the proportion of "fines" required to perform the pipette portion of the grain size analysis
- W Weight of sample in some pipette aliquots was below the level required for accurate weighting

SAMPLE RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *[Signature]*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Client ID: 1406053-01
ARI ID: 14-11372 YN65A

Analyte	Date Batch	Method	Units	RL	Sample
Sulfide	06/13/14 061314#1	SM4500-S2D	mg/L	0.500	2.21
Sulfite	06/12/14 061214#1	SM4500-SO3B	mg/L	1.5	4.3

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *AD*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Client ID: 1406053-02
ARI ID: 14-11373 YN65B

Analyte	Date Batch	Method	Units	RL	Sample
Sulfide	06/13/14 061314#1	SM4500-S2D	mg/L	0.500	1.73
Sulfite	06/12/14 061214#1	SM4500-SO3B	mg/L	1.5	6.3

RL Analytical reporting limit
U Undetected at reported detection limit

MS/MSD RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *W*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Analyte	Method	Date	Units	Sample	Spike	Spike Added	Recovery
ARI ID: YN65A Client ID: 1406053-01							
Sulfide	SM4500-S2D	06/13/14	mg/L	2.21	19.1	16.0	105.6%

REPLICATE RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *W*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Analyte	Method	Date	Units	Sample	Replicate(s)	RPD/RSD
ARI ID: YN65A		Client ID: 1406053-01				
Sulfide	SM4500-S2D	06/13/14	mg/L	2.21	2.48	11.5%
Sulfite	SM4500-SO3B	06/12/14	mg/L	4.3	4.4	2.3%

LAB CONTROL RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *AD*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: NA
Date Received: NA

Analyte/Method	QC ID	Date	Units	LCS	Spike Added	Recovery
Sulfide SM4500-S2D	ICVL	06/13/14	mg/L	0.508	0.500	101.6%
Sulfite SM4500-SO3B	ICVL	06/12/14	mg/L	4.6	5.0	92.0%

METHOD BLANK RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *AD*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: NA
Date Received: NA

Analyte	Method	Date	Units	Blank	ID
Sulfide	SM4500-S2D	06/13/14	mg/L	< 0.050 U	
Sulfite	SM4500-SO3B	06/12/14	mg/L	< 1.5 U	

SULFITE BENCHSHEET (IODOMETRIC TITRATION)				
SM 4500-SO3 B		Date/Time:	6/12/2014	
		ANALYST:	RR	
Potassium iodate-iodide Titrant				
Enter grams KIO3 added to make up Standard Titrant. If using a previously prepared solution, enter the weight data from the Standard Prep Log.				
ARI # C002311		Date prepared	6/12/2014	
grams KIO3 =	0.4462	volume (L) =	1.0	
		Normality (eq/L) =	0.0125	
Laboratory Control Sample				
grams Na2SO3 =	0.3936	volume (mL) =	500	mg/L 500
LCS =	0.5	mL Stk to	50	5.0
Calculation:				
mg SO3 /L = [(mL KIO3 sample - mL KIO3 blank) X N KIO3 X 40,000] / mL sample				
Valid sample titration must be > mean blank + 3 stdev				
SAMPLE DATA		Buret ID:		
	TITRATION DATA			
SAMPLE ID	VOLUME SAMPLE (mL)	VOLUME KI-KIO3 (mL)	Titration Result (mg/L SO3)	Sulfite reported as (mg/L SO3)
BLANK	50	0.08		
BLANK	50	0.07	stdev =	0.010
BLANK	50	0.06	RSD =	14.3%
				OK!
MEAN BLANK	50	0.07		0.7
<i>blimit = mean+ (3 stdev) = 0.10</i>			<i>DL = 1.0</i>	
LCS	50	0.53	4.60	92.1 % Rec
YN65 A2	50	0.50	4.30	4.3
YN65 A2 dup	50	0.51	4.40	4.4
			RPD =	2.3 %
YN65 B2	50	0.70	6.31	6.3

W
6.17.14

SULFIDE BENCHSHEET (Spectrophotometric, EPA 376.2)				Date / Time		Analyst					
Aqueous Samples				Distillation		NA					
				Finish		6/13/14 17:02					
If distilled, specify procedure: NA				zinc acetate: C001955							
1. Standardization of sodium thiosulfate titrant				Buret used for titrations: S2							
Thiosulfate ID: C001106				Titration of bi-iodate with thiosulfate							
Bi-iodate ID: C000374											
Stock bi-iodate = 0.8116 grams to 1000 mL				ml bi-iodate = 3.00 3.00 3.00							
Normality = 0.025				ml thiosulfate = 3.11 3.10 3.07							
Normality thiosulfate = (mL bi-iodate*normality) / mL thiosulfate =				0.024 0.024 0.024							
2. Normality of Iodine				Titration of Iodine with thiosulfate							
Iodine ID: C000565				mL Iodine = 5.00 5.00 5.00							
				mL thiosulfate = 5.05 5.05 5.00							
Normality iodine = (mL thiosulfate*normality) / mL iodine =				0.024 0.024 0.024							
3. Standardization of sodium sulfide stock				Titration of standard with Thiosulfate							
Stock ID = C002319				mL Standard = 1.00 1.00 1.00							
Approx conc in 100ml				mL Iodine = 3.00 3.00 3.00							
g Na2S 0.5153 mg/mL = 0.688				mL thiosulfate = 0.96 0.97 0.93							
Sulfide (mg/mL) = (((mL iodine*ni)-(mL thio *nthio))*16) / mL standard =				0.80 0.79 0.81							
Intermediate Standard				mL required for for 0.025 mg/mL 7.8							
Add 7.8 ml stk to 250 ml 0.2 N ZnOAc =				0.025 mg/mL							
5.0 Calibration Standard Curve				spectrophotometer used:							
Volume Intermediate (ml)			FINAL VOLUME (ml)			CONC (mg S/L)			REGRESSION DATA		
			ABSORBANCE @ 650 nm						intercept 0.006		
			1 2 Avg						slope 0.705		
0.00			50 0.000			0.000			r= 0.9997		
0.10			50 0.050			0.039			Comment: Calibration OK!		
0.25			50 0.125			0.093			maxabs = 0.705		
0.50			50 0.250			0.188					
1.00			50 0.500			0.367					
2.00			50 1.000			0.705					
Calib Verif Std = 1.0 ml int to 50 ml ZnOAc = 0.500 mg/L				Distillation Prep Std = 3.0 ml Stk to 300 = 8.010 mg/L							
SAMPLE DATA				enter dilution factor as ml final/mL sample							
DISTILL DATA		SPECTROPHOTOMETRIC DATA				SAMPLE DATA					
SAMPLE ID	Sample Volume	Distill Volume (mL)	Dilution factor	ABS @ 650 nm	BKG ABS	Regressed Conc (mg S/L)	Final Conc mg S/L				
Cal Blk		n/a	1.00	0.000		-0.008	< 0.05	OK!			
ICV		n/a	1.00	0.364		0.508	0.51	101.63%			
YN65 A1			10.00	0.162		0.221	2.21				
YN65 A1 dup			10.00	0.181		0.248	2.48	RPD=11.5%			
YN65 A1 ms			50.00	0.275		0.382	19.09	105.3%			
Spike at 0.10 ml STK to		5.00 ml sample =		16.02 mg/l							
YN65 B1			10.00	0.128		0.173	1.73				
Prep Blk			1.00	-0.015		-0.030	< 0.05	OK!			
Prep Std			10.00	0.547		0.768	7.68	95.82%			
YN82 A1			1.00	-0.020		-0.037	< 0.05				
YN82 A1 dup			1.00	-0.004		-0.014	< 0.05	NA			
YN82 A1 ms			1.00	0.337		0.470	0.47	94.0%			
Spike at 0.10 ml INT to		5.00 ml sample =		0.50 mg/l							
Cal Blk		n/a	1.00	0.000		-0.008	< 0.05	OK!			
CCV		n/a	1.00	0.363		0.507	0.51	101.34%			

SULFIDE BENCHSHEET (Spectrophotometric, EPA 376.2)

Aqueous Samples Distillation Finish: NA Date / Time: 6/13/14 17:02 Analyst: NA
 If distilled, specify procedure: NA zinc acetate: C001955 Buret used for titrations: S2

1. Standardization of sodium thiosulfate titrant
 Thiosulfate ID: C001106
 Bi-iodate ID: C000374
 Stock bi-iodate = 0.8116 grams to 1000 mL ml bi-iodate = 3.00
 Normality = 0.025 ml thiosulfate = 3.11 3.10 3.07 nthio
 Normality thiosulfate = (mL bi-iodate * normality) / mL thiosulfate =

2. Normality of Iodine
 Iodine ID: C000565
 mL Iodine = 5.00 5.00 5.00
 mL thiosulfate = 5.05 5.05 5.00 ni
 Normality iodine = (mL thiosulfate * normality) / mL iodine =

3. Standardization of sodium sulfide stock
 Stock ID = C002319
 Approx conc in 100ml: 0.5153 mg/mL = 0.688
 g Na2S mL Standard = 1.00 1.00 1.00
 mL iodine = 3.00 3.00 3.00
 mL thiosulfate = 0.96 0.97 0.93 stkconc (mg/mL)
 Sulfide (mg/mL) = {(mL iodine * ni) - (mL thio * nthio)} * 16 / mL standard =

Intermediate Standard
 Add 7.9 ml stk to 250 ml 0.2 N ZnOAc = mg/mL
 mL required for 0.025 mg/mL

5.0 Calibration Standard Curve spectrophotometer used:

Volume Intermediate (ml)	FINAL VOLUME (ml)	CONC (mg S/L)	ABSORBANCE @ 650 nm			REGRESSION DATA
			1	2	Avg	
0.00	50		0.000			intercept slope r= Comment: maxabs =
0.10	50	#VALUE!	0.039			
0.25	50	#VALUE!	0.093			
0.50	50	#VALUE!	0.188			
1.00	50	#VALUE!	0.367			
2.00	50	#VALUE!	0.705			

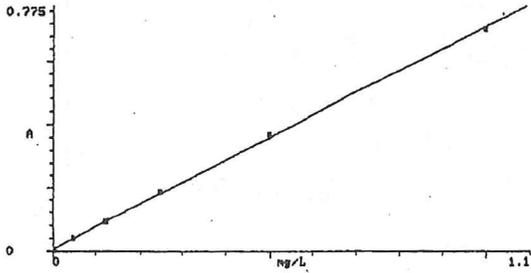
Calib Verif Std = 1.0 ml int to 50 ml ZnOAc = #VALUE! mg/L
 Distillation Prep Std = 3.0 ml Stk to 300 = #VALUE! mg/L

SAMPLE DATA enter dilution factor as ml final/mL sample

SAMPLE ID	DISTILL DATA		SPECTROPHOTOMETRIC DATA			SAMPLE DATA	
	Sample Volume	Distill Volume (mL)	Dilution factor	ABS @ 650 nm	BKG ABS		Regressed Conc (mg S/L)
Cal Blk		n/a	1.00	0.000			
ICV		n/a	1.00	0.364			
YN65 A1			10 1.00	0.112			
YN65 A1 dup			10 1.00	0.181			
YN65 A1 ms			50 1.00	0.275			
Spike at 0.10 ml INT to 5.00 ml sample =				#VALUE!		mg/l	
YN65 B1			10 1.00	0.128			
Prep Blk			1.00	-0.05			
Prep Std			10.00	0.547			
YN82 A1			1.00	0.00			
YN82 A1 dup			1.00	0.004			
YN82 A1 ms			1.00	0.337			
Spike at 0.10 ml INT to 5.00 ml sample =				#VALUE!		mg/l	
Cal Blk		n/a	1.00	0.000			
CCV		n/a	1.00	0.313			
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
Cal Blk		n/a	1.00		0.000		
CCV		n/a	1.00				

TEST SETUP
GENESYS 10 v2.021 2G2G048006

Standard Curve 17:02 13Jun14
 Test Name SULFIDE
 Date Standards Measured 13Jun14
 Wavelength 650nm
 Ref. Wavelength Correction Off
 Curve Fit Linear
 Number of Standards 6
 Units mg/L
 ID# (0=OFF) Off
 Low/High Limits 0.050/1.000
 Statistics Off
 Auto Print On



Curve Fit Linear
 Slope 0.705
 Intercept 0.0059
 Std Dev 0.007
 Corr Coeff 1.000

Conc. mg/L	Abs 650nm
0.000	0.000
0.050	0.039
0.125	0.093
0.250	0.188
0.500	0.367
1.000	0.705

6/13/14
APD

2 0.364

3 0.162

4 0.181

5 0.275

6 0.128

7 -0.015

8 0.547

9 -0.020

10 -0.004

11 0.337

TEST SETUP
GENESYS 10 v2.021 2G2G048006

Advanced A-%T-C 17:03 13Jun14
 Test Name SULFIDE[Saved]
 Measurement Mode Absorbance
 Wavelength 650nm
 Ref. Wavelength Correction Off
 Delay Time (min:sec) 0:00
 ID# (0=OFF) 1
 Low/High Limits 0.000/0.800
 Statistics Off
 Auto Print On

ID#	Abs 650nm
1	0.000

12 0.000

13 0.363



Conventional Laboratory Analyst Notes

ARI Job No.: YN65

Client ID: WDOE

Parameter: SO₃⁻²

Client Project: WDOE 1406053

List problems, concerns, corrective actions and any other pertinent information

Both samples were positive for S²⁻ and were subsequently treated with ZnOAc

Analyst Initials:

RR

Date:

6/12/14

Washington State Department of Ecology (ECY)
Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

July 9, 2014

Project: Black Water – Skagit County

ECY LIMS Work Order #: 1406053

Contract Laboratory: Analytical Resources, Inc.

Contract Lab Sample ID: YN65

Project Officer: Jeanne Tran

Sulfide by Standard Methods 4500-S²⁻ D

Sulfite by Standard Methods 4500-SO₃ B

Enclosed are the results for samples collected on 6/12/2014. If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

**Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist**

Project: Black Water

Analysis: Sulfide and Sulfite

Work Order: 1406053

Project Officer: Jeanne Tran

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	X			
Did sample arrive in a state of proper preservation at contract lab?		X		<ol style="list-style-type: none"> 1. Samples were not packed with frozen ice for transport to the lab. They arrived at a temperature of 23.8°C. All sample results have therefore been qualified as estimates; "J". 2. TestAmerica neglected to add EDTA to the sample container for sulfite analysis. If any metals were present, they would interfere with the determination of sulfite in the absence of EDTA. The results for sulfite should be considered estimated, as it is potentially biased either high or low, depending on what metals are present. All sample results for sulfite have therefore been qualified "J". 3. The analyst recorded the wrong preservation information on the sample receipt paperwork. She confirmed it was a written error. TestAmerica will hand correct the error from "<12" to ">9", and initial and date, to show the samples to be tested for sulfide were at the correct pH. 4. The samples for sulfite were tested for and were confirmed to contain sulfide. Prior to analysis, sulfide was removed from these samples by precipitation with zinc acetate as required by the method.
Are the holding times within acceptable limits for preparation and analysis?		X		<p>Samples for sulfide are to be analyzed within 7 days of collection for regulatory purposes under 40CFR. Sulfite is supposed to be fixed with EDTA and analyzed immediately.</p> <p>Both analyses were conducted within one day of sample collection.</p> <p>Sulfite samples are to be considered estimated and have been qualified "J".</p>
Are all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within acceptable limits for the Initial Calibration (ICAL)?	X			
Are all of the analytes within acceptable limits for the Initial Calibration Verifications (ICV)?	X			
Was a Continuing Calibration Verification (CCV)	X			

analyzed every 10 samples?				
Are all of the analytes for the Continuing Calibration Verification (CCV) within acceptable limits?	X			
Was the Laboratory Control Sample (LCS) spiked with all target analytes and are percent recoveries within quality control (QC) limits?	X			
Is one method blank analyzed per batch of 20 samples or less, and is it free of any positive results?		X		
Were the matrix spike percent recoveries within QC limits?	X			
If a Sample Duplicate was analyzed, is the RPD within QC limits?	X			<20%
Are the results correctly calculated, with proper units and within the linear range of the calibrations?	X			

NA – Not available/Not applicable.

Qualifier Description

- J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- U The analyte was not detected at or above the reported sample quantitation limit.

SAMPLE RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *W*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Client ID: 1406053-01
ARI ID: 14-11372 YN65A

Analyte	Date Batch	Method	Units	RL	Sample
Sulfide	06/13/14 061314#1	SM4500-S2D	mg/L	0.500	2.21 <i>J</i> <i>KF</i>
Sulfite	06/12/14 061214#1	SM4500-SO3B	mg/L	1.5	4.3 <i>J</i> <i>KF</i>

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *AD*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Client ID: 1406053-02
ARI ID: 14-11373 YN65B

Analyte	Date Batch	Method	Units	RL	Sample	
Sulfide	06/13/14 061314#1	SM4500-S2D	mg/L	0.500	1.73	J KF
Sulfite	06/12/14 061214#1	SM4500-SO3B	mg/L	1.5	6.3	J KF

RL Analytical reporting limit
U Undetected at reported detection limit

REPLICATE RESULTS-CONVENTIONALS
 YN65-Washington Department of Ecology



Matrix: Water
 Data Release Authorized: *W*
 Reported: 06/19/14

Project: Black Water
 Event: NA
 Date Sampled: 06/12/14
 Date Received: 06/12/14

Analyte	Method	Date	Units	Sample	Replicate(s)	RPD/RSD
ARI ID: YN65A	Client ID: 1406053-01					
Sulfide	SM4500-S2D	06/13/14	mg/L	2.21 <i>J</i>	2.48 <i>J</i> <i>KF</i>	11.5%
Sulfite	SM4500-SO3B	06/12/14	mg/L	4.3 <i>J</i>	4.4 <i>J</i> <i>KF</i>	2.3%

MS/MSD RESULTS-CONVENTIONALS
YN65-Washington Department of Ecology



Matrix: Water
Data Release Authorized: *W*
Reported: 06/19/14

Project: Black Water
Event: NA
Date Sampled: 06/12/14
Date Received: 06/12/14

Analyte	Method	Date	Units	Sample	Spike	Spike Added	Recovery
ARI ID: YN65A	Client ID: 1406053-01						
Sulfide	SM4500-S2D	06/13/14	mg/L	2.21 <i>J</i>	19.1 <i>J</i>	16.0	105.6%



ARI Job No: YN65

PC: Cheronne
VTSR: 06/12/14

Inquiry Number: NONE
Analysis Requested: 06/13/14
Contact: Feddersen, Karin
Client: Washington Department of Ecology
Logged by: AV
Sample Set Used: Yes-481
Validatable Package: No
Deliverables:

Project #: 1406053
Project: Black Water
Sample Site:
SDG No:
Analytical Protocol: In-house

LOGNUM	CLIENT ID	CN	WAD	NH3	COD	FOG	MET	PHEN	PHOS	TKN	NO23	TOC	S2	TPHD	Fe2+	DMET DOC	FLT	FLT	PARAMETER	ADJUSTED TO	LOT NUMBER	AMOUNT ADDED	DATE/BY
14-11372	FIELD DITCH	>12	>12	<2	<2	<2	<2	<2	<2	<2	<2	<2	>9	<2	<2				SZ PH	SD	024K	2014	6/12/14
YN65A													*						I		6114014	I	ADD
14-11373	SETTLING POND																						
YN65B																							

* Sulfide preserved w/ ZINCAC, lab to adjust Ph.

90 7/10/14
>9
I

ADD 6/12/14

90 7/10/14

W
6.17-14

Methylene Blue, SM4500-S2-D

SULFIDE BENCHSHEET (Spectrophotometric, EPA 376.2)		Date / Time	Analyst				
Aqueous Samples		NA	NA				
Distillation Finish		6/13/14 17:02	APD				
If distilled, specify procedure: NA		zinc acetate: C001955					
1. Standardization of sodium thiosulfate titrant		Buret used for titrations: S2					
Thiosulfate ID: C001106							
BI-Iodate ID: C000374							
Stock bi-Iodate = 0.8116 grams to 1000 mL		ml bi-Iodate = 3.00 3.00 3.00					
Normality = 0.025		ml thiosulfate = 3.11 3.10 3.07					
Normality thiosulfate = (mL bi-Iodate*normality) / mL thiosulfate =		0.024 0.024 0.024					
2. Normality of Iodine		Titration of Iodine with thiosulfate					
Iodine ID: C000585		mL Iodine = 5.00 5.00 5.00					
		mL thiosulfate = 5.05 5.05 5.00					
Normality iodine = (mL thiosulfate*normality) / mL iodine =		0.024 0.024 0.024					
3. Standardization of sodium sulfide stock		Titration of standard with Thiosulfate					
Stock ID = C002319		mL Standard = 1.00 1.00 1.00					
Approx conc in 100ml		mL Iodine = 3.00 3.00 3.00					
g Na2S 0.5153 mg/mL = 0.888		mL thiosulfate = 0.98 0.97 0.93					
Sulfide (mg/mL) = ((mL Iodine*ni)-(mL thio *nthio))*16 / mL standard =		0.80 0.79 0.81					
Intermediate Standard		mL required for for 0.025 mg/mL 7.8					
Add 7.8 ml stk to 250 ml 0.2 N ZnOAc =		0.025 mg/mL					
5.0 Calibration Standard Curve		spectrophotometer used:					
Volume Intermediate (ml)	FINAL VOLUME (ml)	CONC (mg S/L)	ABSORBANCE @ 650 nm	REGRESSION DATA Intercept 0.006 slope 0.705 r= 0.9997 Comment: Calibration OK! maxabs = 0.705			
0.00	50	0.000	1 2 Avg				
0.10	50	0.050	0.039 0.039 0.047				
0.25	50	0.125	0.093 0.093 0.124				
0.50	50	0.250	0.188 0.188 0.258				
1.00	50	0.500	0.367 0.367 0.512				
2.00	50	1.000	0.705 0.705 0.992				
Callb Verif Std = 1.0 ml int to 50 ml ZnOAc = 0.500 mg/L							
Distillation Prep Std = 3.0 ml Stk to 300 = 8.010 mg/L							
SAMPLE DATA		enter dilution factor as ml final/mL sample					
SAMPLE ID	DISTILL DATA		SPECTROPHOTOMETRIC DATA			SAMPLE DATA	
	Sample Volume	Distill Volume (mL)	Dilution factor	ABS @ 650 nm	BKG ABS		Regressed Conc (mg S/L)
Cal Blk	n/a	n/a	1.00	0.000		-0.008	< 0.05 OK!
ICV	n/a	n/a	1.00	0.364		0.508	0.51 101.63%
YN65 A1			10.00	0.162		0.221	2.21
YN65 A1 dup			10.00	0.181		0.248	2.48 RPD=11.5%
YN65 A1 ms			50.00	0.275		0.382	19.09 105.3%
Spike at 0.10 ml STK to		5.00 ml sample =		18.02 mg/l			
YN65 B1			10.00	0.128		0.173	1.73
Prep Blk			1.00	-0.015		-0.030	< 0.05 OK!
Prep Std			10.00	0.547		0.768	7.68 95.82%
YN82 A1			1.00	-0.020		-0.037	< 0.05
YN82 A1 dup			1.00	-0.004		-0.014	< 0.05 NA
YN82 A1 ms			1.00	0.337		0.470	0.47 94.0%
Spike at 0.10 ml INT to		5.00 ml sample =		0.50 mg/l			
Cal Blk			1.00	0.000		-0.008	< 0.05 OK!
CCV			1.00	0.363		0.507	0.61 101.34%

ω = 1/3/14

YN65: 00030REN

Methylene Blue, SM4500-S2-D

SULFIDE BENCHSHEET - (Spectrophotometric, EPA 376.2)		Date / Time	Analyst				
Aqueous Samples		NA	NA				
Distillation Finish		6/13/14 17:02	APD				
If distilled, specify procedure: NA		zinc acetate: C001955					
1. Standardization of sodium thiosulfate titrant		Buret used for titrations: S2					
Thiosulfate ID: C001106							
Bi-iodate ID: C000374		Titration of bi-iodate with thiosulfate					
Stock bi-iodate = 0.8118 grams to 1000 mL	ml bi-iodate = 3.00	3.00	3.00				
Normality = 0.025	ml thiosulfate = 3.11	3.10	3.07				
Normality thiosulfate = (mL bi-iodate * normality) / mL thiosulfate =							
2. Normality of Iodine		Titration of Iodine with thiosulfate					
Iodine ID: C000565		mL Iodine = 5.300					
		mL thiosulfate = 5.05					
Normality iodine = (mL thiosulfate * normality) / mL iodine =		5.300					
		5.05					
		5.02					
3. Standardization of sodium sulfide stock		Titration of standard with Thiosulfate					
Stock ID = C002319		mL Standard = 1.00					
Approx conc in 100ml		mL iodine = 3.00					
g Na2S = 0.5153 mg/mL = 0.888	mL thiosulfate = 0.96	0.97	0.93				
Sulfide (mg/mL) = ((mL iodine * ni) - (mL thio * nthio)) * 16 / mL standard =		stk conc (mg/mL)					
Intermediate Standard		mL required for for 0.025 mg/mL					
Add 7.9 ml stk to 250 ml 0.2 N ZnOAc =	mg/mL						
5.0 Calibration Standard Curve spectrophotometer used:							
Volume Intermediate (ml)	FINAL VOLUME (ml)	CONC (mg S/L)	ABSORBANCE @ 650 nm			REGRESSION DATA	
			1	2	Avg		
0.00	50		0.000			Intercept slope r= Comment: maxabs =	
0.10	50	#VALUE!	0.039				
0.25	50	#VALUE!	0.093				
0.50	50	#VALUE!	0.188				
1.00	50	#VALUE!	0.364				
2.00	50	#VALUE!	0.705				
Calib Verif Std = 1.0 ml int to 50 ml ZnOAc = #VALUE! mg/L							
Distillation Prep Std = 3.0 ml Stk to 300 = #VALUE! mg/L							
SAMPLE DATA enter dilution factor as ml final/mL sample							
SAMPLE ID	DISTILL DATA		SPECTROPHOTOMETRIC DATA			SAMPLE DATA	
	Sample Volume	Distill Volume (mL)	Dilution factor	ABS @ 650 nm	BKG ABS		Regressed Conc (mg S/L)
Cal Blk		n/a	1.00	0.000			
ICV		n/a	1.00	0.364			
YN65 A1			10 1.00	0.162			
YN65 A1 dup			10 1.00	0.161			
YN65 A1 ms			50 1.00	0.275			
Spike at 0.10 ml INT to		5.00 ml sample =		#VALUE! mg/l			
YN65 B1			10 1.00	0.126			
Prep Blk			1.00	-0.05			
Prep Std			10.00	0.547			
YN82 A1			1.00	-0.020			
YN82 A1 dup			1.00	-0.024			
YN82 A1 ms			1.00	0.337			
Spike at 0.10 ml INT to		5.00 ml sample =		#VALUE! mg/l			
Cal Blk		n/a	1.00	0.000			
CCV		n/a	1.00	0.313			
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
			1.00				
Cal Blk		n/a	1.00	0.000			
CCV		n/a	1.00				

YN65: 00021REV 00 =led14

Manchester Environmental Laboratory
7411 Beach Drive E, Port Orchard, Washington 98366

Case Narrative

June 20, 2014

Project: Microbiology Black Water

Work Order: 1406053

Project
Manager: Tran, Jeanne

By: Edlin Limmer, Microbiologist 2 

Summary

The samples were analyzed for fecal coliforms following Standard Methods 9222D.

The analysis requested was evaluated by established regulatory quality assurance guidelines.

Sample Information

The samples were received at the Manchester Laboratory on 6/13/2014. The cooler was received at 11°C, above the proper temperature range of 0°C - <10°C. The samples were qualified as estimates. Two samples were received and assigned laboratory identification numbers 01 and 02.

Holding Times

Holding times used at the Manchester Laboratory follow Standard Methods 20th Edition, 9060B. The samples were analyzed within their hold times.

Replicates

The duplicate relative percent difference with plate counts > 20 counts was within the acceptance limits.

Controls

Pre and post filtering blanks were analyzed with each set of samples. The blanks were negative for growth.

Other Quality Assurance Measures and Issues

J - The organism was positively identified. The associated numerical result is an estimate.

U - The organism was not detected at or above the reported result.

G -Greater than

NC -Not calculated.

Bold - The analyte was present in the sample. (Visual Aid to locate detected organisms on report sheet.)

Please call Edlin Limmer at (360) 871-8810 to further discuss this project.
cc: Project File.

Manchester Environmental Laboratory
7411 Beach Drive E, Port Orchard, Washington 98366

Case Narrative

June 23, 2014

Project: Metals Black Water

Work Order: 1406053

Project
Manager: Tran, Jeanne

By: Dean Momohara

Summary

The laboratory followed EPA 200.7 for the preparation and analysis of trace metals. The samples were also analyzed for sulfur. The sulfur (S) result for samples 01 and 02 were 10.5 ppm and 18.6 ppm, respectively. Since the laboratory is not accredited for S analysis, the results are for informational purposes and should be viewed as estimates.

The samples also contained high levels of silicon (Si), 38.7 ppm and 27.2 ppm in samples 01 and 02, respectively. The levels detected may not be a true indicator of the Si in the samples. Since the preparation used to digest the samples is not suitable for Si, the results should be used for informational purposes and be viewed as estimates.

The analysis requested was evaluated by established regulatory quality assurance guidelines.

Sample Information

The samples were received at the Manchester Laboratory on 6/13/2014. The samples were received in good condition and were properly preserved. Two samples were received and assigned laboratory identification numbers 01 and 02.

Holding Times

The laboratory performed the analysis within its hold time.

Calibration

The instrument was calibrated following the appropriate method. All initial and continuing calibration verification checks were within the acceptance limits. All initial and continuing calibration verification and blank checks were within the acceptance limits. The instrument was calibrated with a NIST traceable standard and verified to be in calibration with a second source NIST traceable standard.

Method Blanks

No analytically significant level of analyte was detected in the method blank associated with these samples.

Laboratory Control Samples

The laboratory control sample recovery was within the acceptance limits.

Replicates

The associated duplicate relative percent difference of samples with concentrations greater than 5 times the reporting limit was within the acceptance limits.

Matrix Spikes

All matrix spike recoveries were within the acceptance limits.

Internal Standards

NA

Other Quality Assurance Measures and Issues

U - The analyte was not detected at or above the reported result.

bold - The analyte was present in the sample. (Visual Aid to locate detected compounds on report sheet.)

Please call Dean Momohara at (360) 871-8808 to further discuss this project.

cc: Project File

**Washington State Department of Ecology
Manchester Environmental Laboratory
Final Analysis Report for
Manganese**

Project Name: Black Water

Project Officer: Tran, Jeanne
Work Order: 1406053
Analyte: Manganese

Prep Method: EPA200.2
Prepared: 06/17/14
Batch ID: B14F131

Analysis Method: EPA200.7
Matrix: Water
Units: mg/L

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	0.868		0.005	0.0003	06/12/14	06/20/14
1406053-02	Settling Pond	1.12		0.005	0.0003	06/12/14	06/20/14

QC Results for Batch ID: B14F131

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F131-BLK1	Blank	0.005	U	0.005	0.0003

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F131-BS1	LCS	107	2.00		2.13	107	85-115		
B14F131-MS1	Matrix Spike	99	2.00	1406053-01	2.85	99	75-125		
B14F131-MSD1	Matrix Spike Dup	101	2.00	1406053-01	2.88	101	75-125	1	20

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Release Date: 6/23/14

Manchester Environmental Laboratory
7411 Beach Drive E, Port Orchard, Washington 98366

Case Narrative

June 24, 2014

Project: General Chemistry Black Water

Work Order: 1406053

Project
Manager: Tran, Jeanne

By: Dean Momohara
D^r

Summary

The laboratory analyzed the samples following Standard Methods (SM) 4500NH3H for ammonia, SM2130 for turbidity, SM4500NO3I for nitrate-nitrite, EPA 300.0 for sulfate, SM4500NB for total persulfate nitrogen and SM5210B for biochemical oxygen demand.

All analyses requested were evaluated by established regulatory quality assurance guidelines.

Sample Information

The samples were received at the Manchester Laboratory on 6/13/2014. The cooler was received at 11°C, above the proper temperature range of 0°C - 6°C. All results were qualified as estimates. The samples were received in good condition and where applicable, were properly preserved. Two samples were received and assigned laboratory identification numbers 01 and 02.

Holding Times

The laboratory performed all analyses within their hold times.

Calibration

The instruments were calibrated following the appropriate methods. All initial and continuing calibration verification checks were within the acceptance limits. All initial and continuing blank checks were within the acceptance limits. The r-value was within the acceptance limits. All standard residuals were within acceptance limits.

The instruments were calibrated with NIST traceable standards and verified to be in calibration with a second source NIST traceable standard. Incubator drying temperatures were monitored before and after drying.

Method Blanks

No analytically significant levels of analyte were detected in the method blanks associated with these samples.

Laboratory Control Samples

All laboratory control sample recoveries were within the acceptance limits.

Replicates

The associated duplicate relative percent differences of samples with concentrations greater than 5 times the reporting limit were within the acceptance limits.

Matrix Spikes

All matrix spike recoveries were within the acceptance limits.

Other Quality Assurance Measures and Issues

U - The analyte was not detected at or above the reported result.

J - The analyte was positively identified. The associated numerical result is an estimate.

bold - The analyte was present in the sample. (Visual Aid to locate detected compounds on report sheet.)

Please call Dean Momohara at (360) 871-8808 to further discuss this project.

cc: Project File

**Washington State Department of Ecology
 Manchester Environmental Laboratory
 Final Analysis Report for
 Biochemical Oxygen Demand five day test**

Project Name: Black Water

**Project Officer: Tran, Jeanne
 Work Order: 1406053
 Analyte: Biochemical Oxygen Demand**

**Method: SM5210B
 Batch ID: B14F110
 Prepared: 06/13/14**

**Matrix: Water
 Units: mg/L**

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	108	J	75		06/12/14	06/13/14
1406053-02	Settling Pond	181	J	112		06/12/14	06/13/14

QC Results for Batch ID: B14F110

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F110-BLK1	Blank	2	U	2	

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F110-BS1	LCS	94	198		187	94	70-130		
B14F110-DUP1	Duplicate	14		1406031-01	14			4	20

Authorized by: DM

Release Date: 6/24/14

Page 1 of 6

**Washington State Department of Ecology
Manchester Environmental Laboratory
Final Analysis Report for
Ammonia**

Project Name: Black Water

**Project Officer: Tran, Jeanne
Work Order: 1406053
Analyte: Ammonia**

**Method: SM4500NH3H
Batch ID: B14F168
Prepared: 06/20/14**

**Matrix: Water
Units: mg/L**

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	1.99	J	0.100	0.014	06/12/14	06/20/14
1406053-02	Settling Pond	2.31	J	0.150	0.021	06/12/14	06/20/14

QC Results for Batch ID: B14F168

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F168-BLK1	Blank	0.010	U	0.010	0.001

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F168-BS1	LCS	100	0.050		0.050	100	80-120		
B14F168-DUP1	Duplicate	0.530		1406036-01	0.508			4	20
B14F168-MS1	Matrix Spike	97	0.050	1406014-09	0.054	97	75-125		

Authorized by: DM

Release Date: 6/24/14 Page 2 of 6

**Washington State Department of Ecology
Manchester Environmental Laboratory
Final Analysis Report for
Nitrite/Nitrate as N**

Project Name: Black Water

**Project Officer: Tran, Jeanne
Work Order: 1406053
Analyte: Nitrate-Nitrite as N**

**Method: SM4500NO3I
Batch ID: B14F122
Prepared: 06/17/14**

**Matrix: Water
Units: mg/L**

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	0.042	J	0.010	0.003	06/12/14	06/17/14
1406053-02	Settling Pond	0.053	J	0.010	0.003	06/12/14	06/17/14

QC Results for Batch ID: B14F122

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F122-BLK1	Blank	0.010	U	0.010	0.003

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F122-BS1	LCS	103	0.198		0.203	103	80-120		
B14F122-DUP1	Duplicate	0.055		1406005-01	0.053			3	20
B14F122-MS1	Matrix Spike	100	0.198	1406004-01	0.269	100	75-125		

Authorized by: _____ *DM*

Release Date: _____ *6/24/14*

Page 3 of 6

**Washington State Department of Ecology
Manchester Environmental Laboratory
Final Analysis Report for
Sulfate**

Project Name: Black Water

**Project Officer: Tran, Jeanne
Work Order: 1406053
Analyte: Sulfate**

**Method: EPA300.0
Batch ID: B14F119
Prepared: 06/16/14**

**Matrix: Water
Units: mg/L**

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	25.6	J	0.30	0.06	06/12/14	06/16/14
1406053-02	Settling Pond	51.2	J	0.30	0.06	06/12/14	06/16/14

QC Results for Batch ID: B14F119

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F119-BLK1	Blank	0.30	U	0.30	0.06

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F119-BS1	LCS	99	5.00		4.94	99	90-110		
B14F119-DUP1	Duplicate	25.5		1406053-01	25.6			0.4	20
B14F119-MS1	Matrix Spike	106	5.00	1406053-01	30.9	106	75-125		

Authorized by: _____

DM

Release Date: _____

6/24/14

Page 4 of 6

**Washington State Department of Ecology
Manchester Environmental Laboratory
Final Analysis Report for
Total Persulfate Nitrogen as N**

Project Name: Black Water

**Project Officer: Tran, Jeanne
Work Order: 1406053
Analyte: Total Persulfate Nitrogen**

**Method: SM4500NB
Batch ID: B14F117
Prepared: 06/16/14**

**Matrix: Water
Units: mg/L**

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	3.65	J	0.125	0.063	06/12/14	06/16/14
1406053-02	Settling Pond	4.93	J	0.125	0.063	06/12/14	06/16/14

QC Results for Batch ID: B14F117

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F117-BLK1	Blank	0.025	U	0.025	0.013

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F117-BS1	LCS	97	0.397		0.386	97	80-120		
B14F117-DUP1	Duplicate	0.192		1406002-23	0.193			0.7	20
B14F117-MS1	Matrix Spike	93	0.397	1406002-24	0.565	93	75-125		

Authorized by: DM

Release Date: 6/24/14

Page 5 of 6

**Washington State Department of Ecology
Manchester Environmental Laboratory
Final Analysis Report for
Turbidity of water**

Project Name: Black Water

**Project Officer: Tran, Jeanne
Work Order: 1406053
Analyte: Turbidity**

**Method: SM2130
Batch ID: B14F118
Prepared: 06/13/14**

**Matrix: Water
Units: NTU**

Sample #	Sample ID	Result	Qualifier	RL	MDL	Collected	Analyzed
1406053-01	Field Ditch	190	J	0.5	0.1	06/12/14	06/13/14
1406053-02	Settling Pond	180	J	0.5	0.1	06/12/14	06/13/14

QC Results for Batch ID: B14F118

Method Blank	Sample ID	Result	Qualifier	RL	MDL
B14F118-BLK1	Blank	0.5	U	0.5	0.1

Sample #	QC Sample	Result	Spike Level	Source Sample	Source Result	%Rec	%Rec Limits	RPD	RPD Limit
B14F118-BS1	LCS	98	18.3		18.0	98	90-110		
B14F118-DUP1	Duplicate	190	J	1406053-02	180	J		5	20

Authorized by: DM

Release Date: 6/24/14 Page 6 of 6

Manchester Environmental Laboratory
7411 Beach Drive E, Port Orchard, Washington 98366

Case Narrative

June 20, 2014

Project: Microbiology Black Water

Work Order: 1406053

Project
Manager: Tran, Jeanne

By: Edlin Limmer, Microbiologist 2 

Summary

The samples were analyzed for fecal coliforms following Standard Methods 9222D.

The analysis requested was evaluated by established regulatory quality assurance guidelines.

Sample Information

The samples were received at the Manchester Laboratory on 6/13/2014. The cooler was received at 11°C, above the proper temperature range of 0°C - <10°C. The samples were qualified as estimates. Two samples were received and assigned laboratory identification numbers 01 and 02.

Holding Times

Holding times used at the Manchester Laboratory follow Standard Methods 20th Edition, 9060B. The samples were analyzed within their hold times.

Replicates

The duplicate relative percent difference with plate counts > 20 counts was within the acceptance limits.

Controls

Pre and post filtering blanks were analyzed with each set of samples. The blanks were negative for growth.

Other Quality Assurance Measures and Issues

- J - The organism was positively identified. The associated numerical result is an estimate.
- U - The organism was not detected at or above the reported result.
- G -Greater than
- NC -Not calculated.
- Bold** - The analyte was present in the sample. (Visual Aid to locate detected organisms on report sheet.)

Please call Edlin Limmer at (360) 871-8810 to further discuss this project.
cc: Project File.

