

Table of Contents: ARI Job AYR6

Client: Apex Labs

Project: A6D0013

Page From: Page To:

| | | |
|---|-----------|-----------|
| Inventory Sheet | | |
| Cover Letter | <u>1</u> | <u>1</u> |
| Chain of Custody Documentation | <u>2</u> | <u>5</u> |
| Case Narrative, Data Qualifiers, Control Limits | <u>6</u> | <u>12</u> |
| General Chemistry Analysis | | |
| Report and Summary QC Forms | <u>13</u> | <u>23</u> |
| General Chemistry Raw Data | | |
| Analyst Notes and Raw Data | <u>24</u> | <u>33</u> |

A
Signature

May-26-2016
Date



Analytical Resources, Incorporated
Analytical Chemists and Consultants

April 14, 2016

Philip Nerenberg
Apex Laboratories
12232 SW Garden Place
Tigard, OR 97223

RE: Project: A6D0013
ARI Job No.: AYR6

Dear Mr. Nerenberg:

Please find enclosed the original Chain of Custody records (COCs), sample receipt documentation, and the final data for the samples from the project referenced above.

Sample receipt information and analytical details are addressed in the Case Narrative.

An electronic copy of this report and all supporting raw data will be kept on file at ARI. Should you have any questions or concerns, please feel free to call me at your convenience.

Respectfully,
ANALYTICAL RESOURCES, INC.

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

cc: eFile: AYR6

Enclosures

Chain of Custody Documentation

ARI Job ID: AYR6

KF 4/4/16

Ayrb

SUBCONTRACT ORDER

Apex Laboratories

A6D0013

SENDING LABORATORY:

Apex Laboratories
12232 S.W. Garden Place
Tigard, OR 97223
Phone: (503) 718-2323
Fax: (503) 718-0333
Project Manager: Philip Nerenberg

RECEIVING LABORATORY:

Analytical Resources, INC
4611 S. 134th Place
Tukwila, WA 98168
Phone : (206) 695-6200
Fax: (206) 695-6201

NDP Sediment-003 (0-0.5)

Sample Name: 5237-160331-NDP-SED003 Sedimen Sampled: 03/31/16 10:35 (A6D0013-02)

| Analysis | Due | Expires | Comments |
|--|----------------|----------------|----------|
| Sulfide, Total by PSEP (376.2) (SUB) | 04/14/16 17:00 | 04/14/16 10:35 | |
| <i>Containers Supplied:</i> (E)4 oz Glass Jar | | | |

NDP Sediment-002 (0-0.5)

Sample Name: 5237-160331-NDP-SED002 Sedimen Sampled: 03/31/16 10:45 (A6D0013-04)

| Analysis | Due | Expires | Comments |
|--|----------------|----------------|----------|
| Sulfide, Total by PSEP (376.2) (SUB) | 04/14/16 17:00 | 04/14/16 10:45 | |
| <i>Containers Supplied:</i> (G)4 oz Glass Jar | | | |

NDP Sediment-001 (0-0.5)

Sample Name: 5237-160331-NDP-SED001 Sedimen Sampled: 03/31/16 11:00 (A6D0013-06)

| Analysis | Due | Expires | Comments |
|--|----------------|----------------|----------|
| Sulfide, Total by PSEP (376.2) (SUB) | 04/14/16 17:00 | 04/14/16 11:00 | |
| <i>Containers Supplied:</i> (G)4 oz Glass Jar | | | |

NDP Sediment-005 (0-0.5)

Sample Name: 5237-160331-NDP-SED005 Sedimen Sampled: 03/31/16 11:00 (A6D0013-08)

| Analysis | Due | Expires | Comments |
|--|----------------|----------------|----------|
| Sulfide, Total by PSEP (376.2) (SUB) | 04/14/16 17:00 | 04/14/16 11:00 | |
| <i>Containers Supplied:</i> (G)4 oz Glass Jar | | | |

Standard TAT

| | | | |
|--------------------|------|--------------------|---------------|
| Released By | Date | Received By | Date |
| <i>Missy Kye</i> | | <i>[Signature]</i> | 4-5-16 @ 1058 |
| Released By | Date | Received By | Date |
| <i>[Signature]</i> | | <i>[Signature]</i> | |

UPS (Shipper)

UPS (Shipper)

AYR6

SUBCONTRACT ORDER

Apex Laboratories

A6D0013

NDP Sediment-004(0-0.5)

Sample Name: 5237-160331-NDP-SED004 Sedimen Sampled: 03/31/16 11:40 (A6D0013-10)

| Analysis | Due | Expires | Comments |
|---|----------------|----------------|----------|
| Sulfide, Total by PSEP (376.2) (SUB) | 04/14/16 17:00 | 04/14/16 11:40 | |
| Containers Supplied: (G)4 oz Glass Jar | | | |

NDP Embankment (0-3.5)

Sample Name: 5237-160331-NDP-EMB001 Soil Sampled: 03/31/16 14:40 (A6D0013-12)

| Analysis | Due | Expires | Comments |
|---|----------------|----------------|----------|
| Sulfide, Total by PSEP (376.2) (SUB) | 04/14/16 17:00 | 04/14/16 14:40 | |
| Containers Supplied: (E)4 oz Glass Jar | | | |

Released By: Missie Kera Date: Received By: UPS (Shipper) Date: 4-5-16 @ 1058

Released By: UPS (Shipper) Date: Received By: [Signature] Date: [Signature]

AYR6 : 00004



Cooler Receipt Form

ARI Client: Apex
 COC No(s): _____ (NA)
 Assigned ARI Job No: AYR6
 Preliminary Examination Phase:

Project Name: _____
 Delivered by: Fed-Ex UPS Courier Hand Delivered Other: _____
 Tracking No: 12X4720R1393820531 NA

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: 1.6
 If cooler temperature is out of compliance fill out form 00070F
 Temp Gun ID#: D005276

Cooler Accepted by: TR Date: 4-5-16 Time: 1058

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

Samples Logged by: TR Date: 4-5-16 Time: TR 1058 1143

**** 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:

By: _____ Date: _____

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

Case Narrative, Data Qualifiers, Control Limits

ARI Job ID: AYR6



Case Narrative

Client: Apex Laboratories
Project: A6D0013
ARI Job No.: AYR6

Sample Receipt

Analytical Resources, Inc. (ARI) accepted six soil samples on April 5, 2016 under ARI job AYR6. The cooler temperature measured by IR thermometer following ARI SOP was 1.6°C. For further details regarding sample receipt, please refer to the Cooler Receipt Form.

The samples were analyzed for parameters as requested.

Sulfide by SM4500-S2

Sulfide sample volumes were preserved upon receipt.

There were no irregularities with this analysis.

Sample ID Cross Reference Report



ARI Job No: AYR6
Client: Apex Labs
Project Event: A6D0013
Project Name: N/A

| Sample ID | ARI Lab ID | ARI LIMS ID | Matrix | Sample Date/Time | VTSR |
|---------------------------|------------|-------------|----------|------------------|----------------|
| 1. 5237-160331-NDP-SED003 | AYR6A | 16-5494 | Sediment | 03/31/16 10:35 | 04/05/16 10:58 |
| 2. 5237-160331-NDP-SED002 | AYR6B | 16-5495 | Sediment | 03/31/16 10:45 | 04/05/16 10:58 |
| 3. 5237-160331-NDP-SED001 | AYR6C | 16-5496 | Sediment | 03/31/16 11:00 | 04/05/16 10:58 |
| 4. 5237-160331-NDP-SED005 | AYR6D | 16-5497 | Sediment | 03/31/16 11:00 | 04/05/16 10:58 |
| 5. 5237-160331-NDP-SED004 | AYR6E | 16-5498 | Sediment | 03/31/16 11:40 | 04/05/16 10:58 |
| 6. 5237-160331-NDP-EMB001 | AYR6F | 16-5499 | Soil | 03/31/16 14:40 | 04/05/16 10:58 |



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).



- 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)**



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

Analytical Method Information

Printed: 05/26/2016 12:58 pm

Sulfide, SM 4500-S2 D-0, Solid (PSEP) in Solid (SM 4500-S2 D-00)

Preservation: ZnOAc, Cool <6°C

Container: Glass WM, Clear, 2 oz

Amount Required: 100 g

Hold Time: 7 days

| Analyte | MDL | Reporting Limit | Surrogate %Rec | Duplicate RPD | ----Matrix Spike---- %Rec | RPD | --Blank Spike / LCS-- %Rec | RPD |
|---------|--------|-----------------|----------------|---------------|---------------------------|-----|----------------------------|-----|
| Sulfide | 0.0750 | 0.500 mg/kg | | 20 | 75-125 | | 75-125 | 20 |

General Chemistry Analysis
Report and Summary QC Forms

ARI Job ID: AYR6

SAMPLE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Sediment
Data Release Authorized: ()
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

Client ID: 5237-160331-NDP-SED003
ARI ID: 16-5494 AYR6A

| Analyte | Date | Method | Units | RL | Sample |
|------------------------|----------------------|------------|---------|------|----------|
| Preserved Total Solids | 04/06/16 040616#1 | SM2540G | Percent | 0.01 | 72.84 |
| Sulfide | 04/05/16 040516#1 | SM4500-S2D | mg/kg | 1.26 | < 1.26 U |

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Sediment
Data Release Authorized: *AW*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

Client ID: 5237-160331-NDP-SED002
ARI ID: 16-5495 AYR6B

| Analyte | Date | Method | Units | RL | Sample |
|------------------------|----------------------|------------|---------|------|----------|
| Preserved Total Solids | 04/06/16 040616#1 | SM2540G | Percent | 0.01 | 70.95 |
| Sulfide | 04/05/16 040516#1 | SM4500-S2D | mg/kg | 1.28 | < 1.28 U |

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Sediment
Data Release Authorized: *AW*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

Client ID: 5237-160331-NDP-SED001
ARI ID: 16-5496 AYR6C

| Analyte | Date | Method | Units | RL | Sample |
|------------------------|----------------------|------------|---------|------|----------|
| Preserved Total Solids | 04/06/16 040616#1 | SM2540G | Percent | 0.01 | 76.13 |
| Sulfide | 04/05/16 040516#1 | SM4500-S2D | mg/kg | 1.27 | < 1.27 U |

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Sediment
Data Release Authorized: *AD*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

Client ID: 5237-160331-NDP-SED005
ARI ID: 16-5497 AYR6D

| Analyte | Date | Method | Units | RL | Sample |
|------------------------|----------------------|------------|---------|------|--------|
| Preserved Total Solids | 04/06/16 040616#1 | SM2540G | Percent | 0.01 | 52.23 |
| Sulfide | 04/05/16 040516#1 | SM4500-S2D | mg/kg | 1.74 | 22.3 |

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Sediment
Data Release Authorized: (A)
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

Client ID: 5237-160331-NDP-SED004
ARI ID: 16-5498 AYR6E

| Analyte | Date | Method | Units | RL | Sample |
|------------------------|----------------------|------------|---------|------|--------|
| Preserved Total Solids | 04/06/16 040616#1 | SM2540G | Percent | 0.01 | 45.95 |
| Sulfide | 04/05/16 040516#1 | SM4500-S2D | mg/kg | 19.6 | 215 |

RL Analytical reporting limit
U Undetected at reported detection limit

SAMPLE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Soil
Data Release Authorized: *U*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

Client ID: 5237-160331-NDP-EMB001
ARI ID: 16-5499 AYR6F

| Analyte | Date | Method | Units | RL | Sample |
|------------------------|----------------------|------------|---------|------|----------|
| Preserved Total Solids | 04/06/16 040616#1 | SM2540G | Percent | 0.01 | 67.54 |
| Sulfide | 04/06/16 040616#1 | SM4500-S2D | mg/kg | 1.35 | < 1.35 U |

RL Analytical reporting limit
U Undetected at reported detection limit

MS/MSD RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Soil
Data Release Authorized: *AK*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

| Analyte | Date | Units | Sample | Spike | Spike Added | Recovery |
|---|----------|-------|--------|-------|-------------|----------|
| ARI ID: AYR6F Client ID: 5237-160331-NDP-EMB001 | | | | | | |
| Sulfide | 04/06/16 | mg/kg | < 1.35 | 180 | 200 | 90.0% |

REPLICATE RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Sediment
Data Release Authorized: (u)
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: 03/31/16
Date Received: 04/05/16

| Analyte | Date | Units | Sample | Replicate (s) | RPD/RSD |
|--|----------|---------|--------|---------------|---------|
| ARI ID: AYR6A Client ID: 5237-160331-NDP-SED003 | | | | | |
| Preserved Total Solids | 04/06/16 | Percent | 72.84 | 75.09 | 3.0% |
| ARI ID: AYR6F Client ID: 5237-160331-NDP-EMB001 | | | | | |
| Sulfide | 04/06/16 | mg/kg | < 1.35 | < 1.44 | NA |

METHOD BLANK RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Soil
Data Release Authorized: *NO*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: NA
Date Received: NA

| Analyte | Date | Units | Blank | QC ID |
|------------------------|----------|---------|----------|-------|
| Preserved Total Solids | 04/06/16 | Percent | < 0.01 U | ICB |
| Sulfide | 04/05/16 | mg/kg | < 0.05 U | PREP |
| | 04/06/16 | | < 0.05 U | PREP |

LAB CONTROL RESULTS-CONVENTIONALS
AYR6-Apex Labs



Matrix: Soil
Data Release Authorized: *QO*
Reported: 05/26/16

Project: NA
Event: A6D0013
Date Sampled: NA
Date Received: NA

| Analyte/Method | QC ID | Date | Units | LCS | Spike Added | Recovery |
|----------------|-------|----------|-------|------|-------------|----------|
| Sulfide | PREP | 04/05/16 | mg/kg | 7.25 | 7.19 | 100.8% |
| SM4500-S2D | PREP | 04/06/16 | | 7.28 | 7.19 | 101.3% |

General Chemistry Raw Data
Analyst Notes and Raw Data

ARI Job ID: AYR6



Analytical Resources,
Incorporated
Analytical Chemists and
Consultants

TOTAL / VOLATILE SOLIDS (TS/TVS) BENCHSHEET

(2n OAc Preserved)

| Analyst: <u>CA/Juw</u> | | Date: <u>4-6-16</u> | Oven ID: <u>82</u> | Muffle ID: <u>1715</u> | Elapsed Time (> 12 Hrs): | Balance ID: 1123230597 |
|------------------------|--------|---------------------|--------------------|---|--------------------------|------------------------|
| Time in Oven: | | Time Out of Oven: | | TVS (mg/kg dry weight) calculated as: Final Ash Weight (g) = (Minimum Ash Weight - Tare Weight) TVS (mg/kg) = [(Dry Weight - Ash Weight) / (Dry Weight) * 1,000,000 If Ash Weight > Dry Weight then "Check for Error" If Dry Weight - Ash Weight < 0.001 < (1/Dry Weight) * 1,000,000 | | |
| Sample ID | Dish # | CV-02 | CV-02 | CV-02 | Dry Weight | Ash Weight 550°C |
| | | CV-02 | CV-02 | CV-02 | grams | |
| BLANK | 1 | | | | | |
| AYRS A1 | 2 | 1.0884 | 1.0883 | | | |
| AYRS A1 | 3 | 6.1400 | 4.7988 | | | |
| AYRS B1 | 4 | 6.2070 | 4.8382 | | | |
| AYRS C1 | 5 | 6.5503 | 5.0673 | | | |
| AYRS D1 | 6 | 6.8413 | 5.9087 | | | |
| AYRS E1 | 7 | 6.9246 | 5.1432 | | | |
| AYRS A1 | 8 | 6.4081 | 5.1432 | | | |
| AYRS B1 | 9 | 6.9567 | 5.3658 | | | |
| AYRS C1 | 10 | 6.9817 | 5.5222 | | | |
| AYRS D1 | 11 | 7.4530 | 5.6013 | | | |
| AYRS E1 | 12 | 7.4720 | 5.9606 | | | |
| AYRS F1 | 13 | 6.4109 | 3.863.8743 | | | |
| AYRS G1 | 14 | 7.2368 | 3.9429 | | | |
| AYRS H1 | | 7.1163 | 5.1792 | | | |
| <i>4-6-16</i> | | | | | | |

AYRS: 00025

Conventional Distillation and Digestion Log



Method:

Sulfide, PSEP, Solid

Matrix: Soil

Analyst: *CDJ NW*

Prep Date, Time: 4-5-16 11:30

Sample Preparation Log

Reagents, Equipment

HCl+Al: _____
 pH Indicator: D003923
 0.2N ZnOAc: D004538
 Balance: 19350128
 Sulfide Stock: E000991

Step By Step

5 grams sample
 Fill traps to line with 0.2N ZnOAc
 LCS and MS get 1mL S2 Stock
 100mL dispersing water
 ~5mL HCl+Al to pH < 3 by
 Bromophenol Blue Indicator
 60 minutes at 90C
 Decant to 100 mL with ZnOAc

| Lab Number | Name | Sample Amount (g/mL) | Final Volume (mL) | Spike ID | Spike Volume (µL) |
|------------|--------|----------------------|-------------------|----------|-------------------|
| PB | | 100 mL | 100 | | |
| LCS | | ↓ | | E000991 | 1000 |
| AY07 | A' | 5.072 | | | |
| | A' dup | 5.285 | | | |
| | A' ms | 5.412 | | E000991 | 1000 |
| | B' | 5.334 | | | |
| | C' | 5.330 | | | |
| | D' | 5.157 | | | |
| | E' | 5.105 | | | |
| | F' | 5.391 | | | |
| | G' | 5.392 | | | |
| | H' | 5.156 | | | |
| | I' | 5.283 | | | |
| | J' | 5.481 | | | |
| AYR5 | A' | 5.579 | | | |
| | B' | 5.091 | | | |
| | C' | 5.100 | | | |
| | D' | 5.448 | | | |
| | E' | 5.251 | | | |
| AYR6 | A' | 5.439 | | | |
| | B' | 5.524 | | | |
| | C' | 5.194 | | | |
| | D' | 5.504 | | | |
| | E' | 5.547 | ↓ | | |

4-6-16 *NW*

Revision: 0005 7/1/2015

Conventionals Distillation and Digestion Log



Method:

Sulfide, PSEP, Solid

Matrix:

Water

Analyst:

NA

Prep Date, Time:

4-6-16 11:15

Sample Preparation Log

Reagents, Equipment

HCl+Al: D004196
 pH Indicator: D003923
 0.2N ZnOAc: D004538
 Balance: 19350128
 Sulfide Stock: E000991

Step By Step

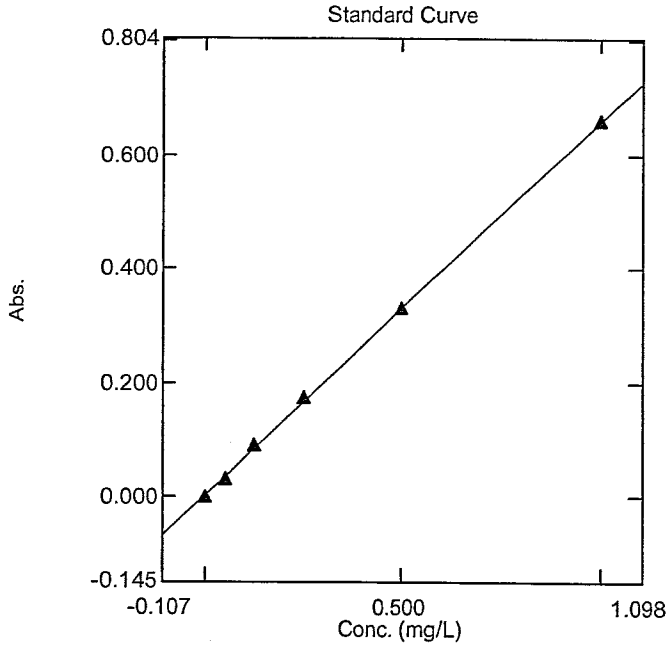
5 grams sample
 Fill traps to line with 0.2N ZnOAc
 LCS and MS get 1mL S2 Stock
 100mL dispersing water
 ~5mL HCl+Al to pH < 3 by
 Bromophenol Blue Indicator
 60 minutes at 90C
 Decant to 100 mL with ZnOAc

| Lab Number | Name | Sample Amount (g/mL) | Final Volume (mL) | Spike ID | Spike Volume (µL) |
|------------|------|----------------------|-------------------|----------|-------------------|
| PB | | | 100 | | |
| LCS | | | ↓ | E000991 | 1000 |
| AYR6 F' | | 5.489 | ↓ | | |
| ↓ F'dup | | 5.129 | ↓ | | |
| ↓ F'ms | | 5.308 | 102 | E000991 | 1000 |
| 4-11-16 NA | | | | | |

Revision: 0005 7/1/2015

Quantitative Measurement Report

Data set: Z:\Shimadzu Spec Methods\SULFIDE DATA 2016\Sulfide 040616b nn.pho



Software Information

Software Name: UVProbe
 Version: 2.51
 Mode: Security Mode

Data Information

Filename: Z:\Shimadzu Spec Methods\SULFIDE DATA 2016
 \Sulfide 040616b nn.pho
 Title: NN APD Sulfide 04 06 16
 Analyst: Nhan Nguyen
 Date/Time: 04/11/2016 12:16:51 PM
 Comments: added dilutions

Instrument Information

Instrument Name: CONV-UV-1
 Instrument Type: UV-1800 Series
 Model (S/N): 206-25400-42 (A11455350830)

$x = 1.52269 y - 0.00631642$
 Correlation Coefficient $r^2 = 0.99971$

Standard Table

| | Sample ID | Date | Time | Conc | Abs@650.0 | Comments |
|---|-----------------|-----------|-------------|-------|-----------|----------|
| 1 | Std 1 (Zero) | 04/06/201 | 02:58:03 PM | 0.000 | -0.000 | |
| 2 | Std 2 (0.10 mL) | 04/06/201 | 02:58:29 PM | 0.050 | 0.034 | |
| 3 | Std 3 (0.25 mL) | 04/06/201 | 02:58:50 PM | 0.125 | 0.091 | |
| 4 | Std 4 (0.50 mL) | 04/06/201 | 02:59:15 PM | 0.250 | 0.174 | |
| 5 | Std 5 (1.00 mL) | 04/06/201 | 02:59:38 PM | 0.500 | 0.331 | |
| 6 | Std 6 (2.00 mL) | 04/06/201 | 03:00:04 PM | 1.000 | 0.660 | |
| 7 | | | | | | |

Quantitative Measurement Report

Data set: Z:\Shimadzu Spec Methods\SULFIDE DATA 2016\Sulfide 040616b nn.pho

Sample Table

| | Sample ID | Date | Time | Conc | Abs@650.0 | DF | Comments |
|----|-------------|------------|-------------|--------|-----------|-------|----------|
| 1 | ICB | 04/06/2016 | 03:01:10 PM | -0.012 | -0.004 | 1.000 | |
| 2 | ICV | 04/06/2016 | 03:01:42 PM | 0.505 | 0.336 | 1.000 | |
| 3 | AYN4 A1 | 04/06/2016 | 03:20:22 PM | 0.022 | 0.019 | 1.000 | |
| 4 | AYN4 A1 Dup | 04/06/2016 | 03:21:07 PM | 0.024 | 0.020 | 1.000 | |
| 5 | AYN4 A1 MS | 04/06/2016 | 03:21:48 PM | 0.349 | 0.233 | 1.000 | |
| 6 | AYN4 B1 | 04/06/2016 | 03:24:12 PM | 0.015 | 0.014 | 1.000 | |
| 7 | AYN4 C1 | 04/06/2016 | 03:25:32 PM | -0.026 | -0.013 | 1.000 | |
| 8 | AYN4 D1 | 04/06/2016 | 03:26:18 PM | -0.026 | -0.013 | 1.000 | |
| 9 | AYN4 E1 | 04/06/2016 | 03:28:02 PM | -0.026 | -0.013 | 1.000 | |
| 10 | AYN4 A1 MS2 | 04/06/2016 | 03:42:33 PM | 0.376 | 0.251 | 1.000 | |
| 11 | AYN4 A1 MS3 | 04/06/2016 | 04:00:15 PM | 0.382 | 0.255 | 1.000 | |
| 12 | CCB | 04/06/2016 | 04:02:47 PM | -0.013 | -0.004 | 1.000 | |
| 13 | CCV | 04/06/2016 | 04:03:25 PM | 0.475 | 0.316 | 1.000 | |
| 14 | ICB 2 | 04/06/2016 | 04:26:42 PM | -0.001 | 0.003 | 1.000 | |
| 15 | ICV 2 | 04/06/2016 | 04:27:12 PM | 0.494 | 0.329 | 1.000 | |
| 16 | PREPBLANK | 04/06/2016 | 04:28:11 PM | -0.001 | 0.004 | 1.000 | |
| 17 | LCS 2 | 04/06/2016 | 04:28:47 PM | 0.719 | 0.476 | 10.00 | |
| 18 | AYO6 A1 | 04/06/2016 | 04:31:06 PM | -0.004 | 0.002 | 1.000 | |
| 19 | AYO6 A1 DU | 04/06/2016 | 04:32:06 PM | -0.004 | 0.001 | 1.000 | |
| 20 | AYO6 A1 MS | 04/06/2016 | 04:32:58 PM | 0.223 | 0.151 | 10.00 | |
| 21 | AYO6 B1 | 04/06/2016 | 04:35:24 PM | -0.010 | -0.002 | 1.000 | |
| 22 | AYO6 C1 | 04/06/2016 | 04:36:26 PM | -0.009 | -0.002 | 1.000 | |
| 23 | AYO6 D1 | 04/06/2016 | 04:37:04 PM | -0.012 | -0.004 | 1.000 | |
| 24 | AYO6 E1 | 04/06/2016 | 04:37:51 PM | -0.012 | -0.003 | 1.000 | |
| 25 | AYO6 F1 | 04/06/2016 | 04:38:36 PM | -0.016 | -0.007 | 1.000 | |
| 26 | CCB 2 | 04/06/2016 | 04:39:08 PM | -0.049 | -0.028 | 1.000 | |
| 27 | CCV 2 | 04/06/2016 | 04:39:51 PM | 0.453 | 0.302 | 1.000 | |
| 28 | AYO6 G1 | 04/06/2016 | 05:05:51 PM | 0.001 | 0.005 | 1.000 | |
| 29 | AYO6 H1 | 04/06/2016 | 05:06:46 PM | 0.000 | 0.004 | 1.000 | |
| 30 | AYO6 I1 | 04/06/2016 | 05:07:32 PM | 0.004 | 0.007 | 1.000 | |
| 31 | AYO6 J1 | 04/06/2016 | 05:08:20 PM | 0.058 | 0.042 | 1.000 | |
| 32 | AYO6 K1 | 04/06/2016 | 05:09:04 PM | 0.380 | 0.253 | 1.000 | |
| 33 | AYO5 A1 | 04/06/2016 | 05:09:55 PM | -0.001 | 0.004 | 1.000 | |
| 34 | AYO5 B1 | 04/06/2016 | 05:10:38 PM | 0.044 | 0.033 | 1.000 | |
| 35 | AYO5 C1 | 04/06/2016 | 05:11:33 PM | -0.007 | -0.000 | 1.000 | |
| 36 | AYO5 D1 | 04/06/2016 | 05:12:45 PM | -0.009 | -0.002 | 1.000 | |
| 37 | AYO5 E1 | 04/06/2016 | 05:13:29 PM | -0.012 | -0.004 | 1.000 | |
| 38 | CCB 3 | 04/06/2016 | 05:14:17 PM | -0.041 | -0.023 | 1.000 | |
| 39 | CCV 3 | 04/06/2016 | 05:15:20 PM | 0.471 | 0.313 | 1.000 | |

Quantitative Measurement Report

Data set: Z:\Shimadzu Spec Methods\SULFIDE DATA 2016\Sulfide 040616b nn.pho

Sample Table

| | Sample ID | Date | Time | Conc | Abs@650.0 | DF | Comments |
|----|-------------|------------|-------------|--------|-----------|-------|----------|
| 40 | AYO5 F1 | 04/06/2016 | 05:33:39 PM | -0.001 | 0.004 | 1.000 | |
| 41 | AYO5 G1 | 04/06/2016 | 05:34:26 PM | -0.001 | 0.003 | 1.000 | |
| 42 | AYO5 H1 | 04/06/2016 | 05:35:04 PM | -0.005 | 0.001 | 1.000 | |
| 43 | AYO6 A1 MS2 | 04/06/2016 | 05:36:11 PM | 0.228 | 0.154 | 10.00 | |
| 44 | CCB 4 | 04/06/2016 | 05:40:40 PM | -0.019 | -0.008 | 1.000 | |
| 45 | CCV 4 | 04/06/2016 | 05:41:03 PM | 0.523 | 0.348 | 1.000 | |
| 46 | ICB 3 | 04/06/2016 | 06:05:14 PM | -0.005 | 0.001 | 1.000 | |
| 47 | ICV 3 | 04/06/2016 | 06:06:00 PM | 0.528 | 0.351 | 1.000 | |
| 48 | PREPBLANK | 04/06/2016 | 06:09:18 PM | -0.008 | -0.001 | 1.000 | |
| 49 | LCS3 | 04/06/2016 | 06:09:41 PM | 0.724 | 0.480 | 10.00 | |
| 50 | AYO7 A1 | 04/06/2016 | 06:11:58 PM | -0.028 | -0.014 | 1.000 | |
| 51 | AYO7 A1 DU | 04/06/2016 | 06:12:26 PM | -0.038 | -0.021 | 1.000 | |
| 52 | AYO7 A1 MS | 04/06/2016 | 06:13:14 PM | 0.565 | 0.375 | 10.00 | |
| 53 | AYO7 B1 | 04/06/2016 | 06:15:46 PM | -0.027 | -0.014 | 1.000 | |
| 54 | AYO7 C1 | 04/06/2016 | 06:16:41 PM | -0.022 | -0.011 | 1.000 | |
| 55 | AYO7 D1 | 04/06/2016 | 06:17:03 PM | -0.021 | -0.010 | 1.000 | |
| 56 | AYO7 E1 | 04/06/2016 | 06:17:28 PM | -0.037 | -0.020 | 1.000 | |
| 57 | AYO7 F1 | 04/06/2016 | 06:17:52 PM | -0.022 | -0.010 | 1.000 | |
| 58 | CCB 5 | 04/06/2016 | 06:18:41 PM | -0.033 | -0.018 | 1.000 | |
| 59 | CCV 5 | 04/06/2016 | 06:19:10 PM | 0.482 | 0.320 | 1.000 | |
| 60 | AYO7 G1 | 04/06/2016 | 06:38:00 PM | 0.004 | 0.007 | 1.000 | |
| 61 | AYO7 H1 | 04/06/2016 | 06:38:26 PM | 0.002 | 0.006 | 1.000 | |
| 62 | AYO7 I1 | 04/06/2016 | 06:38:50 PM | 0.003 | 0.006 | 1.000 | |
| 63 | AYO7 J1 | 04/06/2016 | 06:39:18 PM | 0.002 | 0.005 | 1.000 | |
| 64 | AYR5 A1 | 04/06/2016 | 06:39:42 PM | 0.003 | 0.006 | 1.000 | |
| 65 | AYR5 B1 | 04/06/2016 | 06:40:06 PM | 0.002 | 0.006 | 1.000 | |
| 66 | AYR5 C1 | 04/06/2016 | 06:40:35 PM | 0.044 | 0.033 | 1.000 | |
| 67 | AYR5 D1 | 04/06/2016 | 06:41:10 PM | 0.081 | 0.058 | 1.000 | |
| 68 | AYR5 E1 | 04/06/2016 | 06:41:55 PM | 0.000 | 0.004 | 1.000 | |
| 69 | AYR6 A1 | 04/06/2016 | 06:42:17 PM | -0.001 | 0.003 | 1.000 | |
| 70 | CCB 6 | 04/06/2016 | 06:42:43 PM | -0.004 | 0.001 | 1.000 | |
| 71 | CCV 6 | 04/06/2016 | 06:43:05 PM | 0.505 | 0.336 | 1.000 | |
| 72 | AYR6 B1 | 04/06/2016 | 06:43:51 PM | 0.037 | 0.028 | 1.000 | |
| 73 | AYR6 C1 | 04/06/2016 | 06:44:15 PM | -0.002 | 0.003 | 1.000 | |
| 74 | AYR6 D1 | 04/06/2016 | 06:44:39 PM | 0.642 | 0.426 | 1.000 | |
| 75 | AYR6 E1 | 04/06/2016 | 06:45:14 PM | 0.548 | 0.364 | 1.000 | |
| 76 | CCB 7 | 04/06/2016 | 06:45:50 PM | -0.015 | -0.006 | 1.000 | |
| 77 | CCV 7 | 04/06/2016 | 06:46:14 PM | 0.509 | 0.338 | 1.000 | |
| 78 | ICB 4 | 04/06/2016 | 07:05:04 PM | -0.004 | 0.001 | 1.000 | |

Quantitative Measurement Report

Data set: Z:\Shimadzu Spec Methods\SULFIDE DATA 2016\Sulfide 040616b nn.pho

Sample Table

| | Sample ID | Date | Time | Conc | Abs@650.0 | DF | Comments |
|----|-------------|------------|-------------|--------|-----------|-------|----------|
| 79 | ICV 4 | 04/06/2016 | 07:05:27 PM | 0.506 | 0.337 | 1.000 | |
| 80 | PREPBLANK | 04/06/2016 | 07:06:02 PM | -0.005 | 0.001 | 1.000 | |
| 81 | LCS 3 | 04/06/2016 | 07:06:26 PM | 0.727 | 0.482 | 10.00 | |
| 82 | AYR6 F1 | 04/06/2016 | 07:07:22 PM | 0.045 | 0.034 | 1.000 | |
| 83 | AYR6 F1 DUP | 04/06/2016 | 07:07:45 PM | 0.035 | 0.027 | 1.000 | |
| 84 | AYRS F1 MS | 04/06/2016 | 07:08:10 PM | 0.645 | 0.428 | 10.00 | |
| 85 | CCB 8 | 04/06/2016 | 07:09:45 PM | -0.014 | -0.005 | 1.000 | |
| 86 | CCV 8 | 04/06/2016 | 07:10:00 PM | 0.518 | 0.344 | 1.000 | |
| 87 | | | | | | | |

Quantitative Measurement Report

Data set: Z:\Shimadzu Spec Methods\SULFIDE DATA 2016\Sulfide 040616b nn.pho

[Wavelengths]

Wavelength Name: Abs@650.0
Wavelength: 650.00 nm

[Calibration Curve]

Column for Cal. Curve: Abs@650.0
Cal. Curve Type: Multi Point
Cal. Curve Unit: mg/L
Selected Wavelength: Abs@650.0
Calibration Equation: $\text{Conc} = K1 * (\text{Abs}) + K0$
Zero Interception: Not Selected

[Measurement Parameters(Standard)]

Data Acquired by: Instrument
Delay sample read: Disabled
Repeat: Disabled

[Measurement Parameters(Sample)]

Data Acquired by: Instrument
Delay sample read: Disabled
Repeat: Disabled

[Equations]

Equation Name: AdjConc
Equation: $\text{Conc} * \text{DF}$
Units: mg/L

[Pass Fail]

[Method Summary]

Title: Sulfide Colorimetry
Date/Time: 01/06/2016 05:28:50 PM
Comments:
Sample Preparations:

[Instrument Properties]

Instrument Type: UV-1800 Series
Measuring Mode: Absorbance
Slit Width: 1.0 nm
Light Source Change Wavelength: 340.0 nm
S/R Exchange: Normal

[Attachment Properties]

Attachment: None

SULFIDE TITRATION

Buret used for titrations: S2

Standardization of sodium thiosulfate titrant

Thiosulfate ID: D004645 Analyst: CSL / DAN
 Bi-iodate ID: ~~E002371~~ D003541 Date & Time: 4-6-16
 Stock bi-iodate = 0.8118 grams to 1000 mL
 Normality =
 Titration of bi-iodate with thiosulfate

| | | | | |
|-------------------------|------|------|------|-------|
| mL bi-iodate = | 3.00 | 3.00 | 3.00 | |
| mL thiosulfate = | 3.14 | 3.13 | 3.14 | nthio |
| Normality thiosulfate = | | | | |

(mL bi-iodate * normbio) / mL thiosulfate

Normality of Iodine

Iodine ID: E000905 Analyst:
 Titration of iodine with thiosulfate Date & Time:

| | | | | |
|--------------------|------|------|------|----|
| mL iodine = | 3.00 | 3.00 | 3.00 | |
| mL thiosulfate = | 3.16 | 3.10 | 3.07 | ni |
| Normality iodine = | | | | |

(mL thiosulfate * nthio) / mL iodine

Standardization of Sodium Sulfide Stock

Stock ID = E000991 Analyst:
 Approx conc in 60 mL Date & Time:
 g Na2S = 0.4666 mg/mL =
 Titration of standard with thiosulfate

| | | | | |
|-------------------|------|------|------|-----------------|
| mL Standard = | 1.00 | 1.00 | 1.00 | |
| mL iodine = | 3.00 | 3.00 | 3.00 | |
| mL thiosulfate = | 1.22 | 1.20 | 1.21 | stkconc (mg/mL) |
| Sulfide (mg/mL) = | | | | |

{[(mL iodine * ni) - (mL thio * nthio)] * 16} / mL standard
 mL required for for 0.025 mg/mL

Standardization of Sodium Sulfide Stock

Stock ID = Analyst:
 Approx conc in 60 mL Date & Time:
 g Na2S = mg/mL =
 Titration of standard with thiosulfate

| | | | | |
|-------------------|------|------|------|-----------------|
| mL Standard = | 1.00 | 1.00 | 1.00 | |
| mL iodine = | 3.00 | 3.00 | 3.00 | |
| mL thiosulfate = | | | | stkconc (mg/mL) |
| Sulfide (mg/mL) = | | | | |

{[(mL iodine * ni) - (mL thio * nthio)] * 16} / mL standard
 mL required for for 0.025 mg/mL