# Analytical Laboratory

13339 Hagers Ferry Road
Huntersville, NC 28078-7929
McGuire Nuclear Complex - MG03A2
Phone: 980-875-5245 Fax: 980-875-4349

# Order Summary Report

| | |
|---|---|
| **Order Number:** | **J13040282** |
| Project Name: | WWTS - Biweekly (2) |
| Customer Name(s): | Bill K., Ron L., Don S. |
| Customer Address: | 253 Plant Allen Road<br>Belmont, NC 28012 |
| Lab Contact: | Jason C Perkins Phone: 980-875-5348 |
| **Report Authorized By: (Signature)** | Jason C Perkins — Digitally signed by jay perkins DN: cn=jay perkins, o=Analytical Lab, ou, email=jay.perkins@duke-energy.com, c=US Date: 2013.05.07 11:50:27 -04'00' **Date:** 5/7/2013 |

## Program Comments:

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

### Data Flags & Calculations:

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

*Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)*

### Certification:

The Analytical Laboratory holds the following State Certifications : North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## Sample ID's & Descriptions:



| Sample ID | Plant/Station | Collection Date and Time | Collected By | Sample Description |
|---|---|---|---|---|
| 2013008359 | ALLEN | 19-Apr-13 7:15 AM | illegible | FGD Purge Eff |
| 2013008360 | ALLEN | 19-Apr-13 7:24 AM | illegible | EQ Tank Eff |
| 2013008361 | ALLEN | 19-Apr-13 7:24 AM | illegible | BioReactor 1 Inf |
| 2013008362 | ALLEN | 19-Apr-13 7:34 AM | illegible | BioReactor 2 Inf |
| 2013008363 | ALLEN | 19-Apr-13 7:27 AM | illegible | BioReactor 2 Eff |
| 2013008364 | ALLEN | 19-Apr-13 8:45 AM | illegible | Filter Blk |
| 2013008365 | ALLEN | 15-Apr-13 2:15 PM | C. KNOX | TRIP BLANK |

7 Total Samples

# Technical Validation Review

## Checklist:

| | | |
|---|---|---|
| COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures). | ☑ Yes | ☐ No |
| All Results are less than the laboratory reporting limits. | ☐ Yes | ☑ No |
| All laboratory QA/QC requirements are acceptable. | ☑ Yes | ☐ No |

## Report Sections Included:

- ☑ Job Summary Report
- ☑ Sample Identification
- ☑ Technical Validation of Data Package
- ☑ Analytical Laboratory Certificate of Analysis
- ☐ Analytical Laboratory QC Report
- ☑ Sub-contracted Laboratory Results
- ☐ Customer Specific Data Sheets, Reports, & Documentation
- ☐ Customer Database Entries
- ☑ Chain of Custody
- ☑ Electronic Data Deliverable (EDD) Sent Separately

Reviewed By: DBA Account Date: 5/7/2013

# Certificate of Laboratory Analysis



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**Order # J13040282**

Site: FGD Purge Eff — **Sample #: 2013008359**

Collection Date: 19-Apr-13 7:15 AM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **INORGANIC IONS BY IC** | | | | | | | | |
| Bromide | **330** | mg/L | | 10 | 100 | EPA 300.0 | 04/29/2013 13:49 | JAHERMA |
| **MERCURY (COLD VAPOR) IN WATER** | | | | | | | | |
| Mercury (Hg) | **34.8** | ug/L | | 2.5 | 50 | EPA 245.1 | 04/25/2013 14:52 | AGIBBS |
| **TOTAL RECOVERABLE METALS BY ICP** | | | | | | | | |
| Boron (B) | **130** | mg/L | | 0.5 | 10 | EPA 200.7 | 05/01/2013 11:26 | MHH7131 |
| **DISSOLVED METALS BY ICP-MS** | | | | | | | | |
| Selenium (Se) | **1050** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 13:17 | KRICHAR |
| **TOTAL RECOVERABLE METALS BY ICP-MS** | | | | | | | | |
| Arsenic (As) | **187** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Cadmium (Cd) | **< 10** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Chromium (Cr) | **198** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Copper (Cu) | **166** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Nickel (Ni) | **220** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Selenium (Se) | **2390** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Silver (Ag) | **< 10** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| Zinc (Zn) | **301** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:22 | KRICHAR |
| **SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)** | | | | | | | | |
| Vendor Parameter | **Complete** | | | | | Vendor Method | | V_AS&C |

Site: EQ Tank Eff — **Sample #: 2013008360**

Collection Date: 19-Apr-13 7:24 AM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **MERCURY (COLD VAPOR) IN WATER** | | | | | | | | |
| Mercury (Hg) | **29.1** | ug/L | | 2.5 | 50 | EPA 245.1 | 04/25/2013 14:55 | AGIBBS |
| **TOTAL RECOVERABLE METALS BY ICP** | | | | | | | | |
| Boron (B) | **125** | mg/L | | 0.5 | 10 | EPA 200.7 | 05/01/2013 11:30 | MHH7131 |
| **DISSOLVED METALS BY ICP-MS** | | | | | | | | |
| Selenium (Se) | **904** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 13:21 | KRICHAR |

# Certificate of Laboratory Analysis



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**Order # J13040282**

Site: EQ Tank Eff | **Sample #: 2013008360**

Collection Date: 19-Apr-13 7:24 AM | Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **TOTAL RECOVERABLE METALS BY ICP-MS** | | | | | | | | |
| Arsenic (As) | **156** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Cadmium (Cd) | **< 10** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Chromium (Cr) | **171** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Copper (Cu) | **143** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Nickel (Ni) | **194** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Selenium (Se) | **1900** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Silver (Ag) | **< 10** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |
| Zinc (Zn) | **264** | ug/L | | 10 | 10 | EPA 200.8 | 05/02/2013 12:26 | KRICHAR |

Site: BioReactor 1 Inf | **Sample #: 2013008361**

Collection Date: 19-Apr-13 7:24 AM | Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)** | | | | | | | | |
| Vendor Parameter | **Complete** | ug/l | | | | Vendor Method | | V_AS&C |
| **TOTAL RECOVERABLE METALS BY ICP** | | | | | | | | |
| Boron (B) | **83.2** | mg/L | | 0.5 | 10 | EPA 200.7 | 05/01/2013 11:34 | MHH7131 |
| **DISSOLVED METALS BY ICP-MS** | | | | | | | | |
| Selenium (Se) | **207** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 13:24 | KRICHAR |
| **TOTAL RECOVERABLE METALS BY ICP-MS** | | | | | | | | |
| Arsenic (As) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Cadmium (Cd) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Chromium (Cr) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Copper (Cu) | **22.5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Nickel (Ni) | **5.11** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Selenium (Se) | **190** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Silver (Ag) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| Zinc (Zn) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:29 | KRICHAR |
| **SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)** | | | | | | | | |
| Vendor Parameter | **Complete** | | | | | Vendor Method | | V_AS&C |

# Certificate of Laboratory Analysis



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**Order # J13040282**

Site: BioReactor 2 Inf — **Sample #: 2013008362**

Collection Date: 19-Apr-13 7:34 AM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)** | | | | | | | | |
| Vendor Parameter | **Complete** | ug/l | | | | Vendor Method | | V_AS&C |
| **TOTAL RECOVERABLE METALS BY ICP** | | | | | | | | |
| Boron (B) | **73.2** | mg/L | | 0.5 | 10 | EPA 200.7 | 05/01/2013 11:38 | MHH7131 |
| **TOTAL RECOVERABLE METALS BY ICP-MS** | | | | | | | | |
| Arsenic (As) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Cadmium (Cd) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Chromium (Cr) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Copper (Cu) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Nickel (Ni) | **5.25** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Selenium (Se) | **21.8** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Silver (Ag) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |
| Zinc (Zn) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:32 | KRICHAR |

Site: BioReactor 2 Eff — **Sample #: 2013008363**

Collection Date: 19-Apr-13 7:27 AM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **INORGANIC IONS BY IC** | | | | | | | | |
| Bromide | **240** | mg/L | | 10 | 100 | EPA 300.0 | 04/29/2013 14:08 | JAHERMA |
| **Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)** | | | | | | | | |
| Vendor Parameter | **Complete** | ug/l | | | | Vendor Method | | V_AS&C |
| **TOTAL RECOVERABLE METALS BY ICP** | | | | | | | | |
| Boron (B) | **47.9** | mg/L | | 0.5 | 10 | EPA 200.7 | 05/01/2013 11:42 | MHH7131 |
| **TOTAL RECOVERABLE METALS BY ICP-MS** | | | | | | | | |
| Arsenic (As) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Cadmium (Cd) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Chromium (Cr) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Copper (Cu) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Nickel (Ni) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Selenium (Se) | **7.29** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Silver (Ag) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| Zinc (Zn) | **< 5** | ug/L | | 5 | 5 | EPA 200.8 | 05/02/2013 12:36 | KRICHAR |
| **SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)** | | | | | | | | |
| Vendor Parameter | **Complete** | | | | | Vendor Method | | V_AS&C |

# Certificate of Laboratory Analysis



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**Order # J13040282**

Site: BioReactor 2 Eff
Collection Date: 19-Apr-13 7:27 AM

**Sample #:** **2013008363**
Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **TOTAL DISSOLVED SOLIDS** | | | | | | | | |
| TDS | **8700** | mg/L | | 500 | 1 | SM2540C | 04/29/2013 16:11 | TJA7067 |

Site: Filter Blk
Collection Date: 19-Apr-13 8:45 AM

**Sample #:** **2013008364**
Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **DISSOLVED METALS BY ICP-MS** | | | | | | | | |
| Selenium (Se) | **2.89** | ug/L | | 2 | 2 | EPA 200.8 | 05/02/2013 13:14 | KRICHAR |

Site: TRIP BLANK
Collection Date: 15-Apr-13 2:15 PM

**Sample #:** **2013008365**
Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | DF | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|---|
| **TOTAL RECOVERABLE METALS BY ICP** | | | | | | | | |
| Boron (B) | **< 0.05** | mg/L | | 0.05 | 1 | EPA 200.7 | 05/01/2013 11:19 | MHH7131 |
| **TOTAL RECOVERABLE METALS BY ICP-MS** | | | | | | | | |
| Arsenic (As) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Cadmium (Cd) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Chromium (Cr) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Copper (Cu) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Nickel (Ni) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Selenium (Se) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Silver (Ag) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |
| Zinc (Zn) | **< 1** | ug/L | | 1 | 1 | EPA 200.8 | 05/02/2013 12:15 | KRICHAR |

APPLIED SPECIATION AND CONSULTING, LLC

18804 Northcreek Parkway Bothell, WA, 98011
Tel: (425) 483-3300 Fax: (425) 483-9818
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May 6, 2013

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078
(704) 875-5245

Project: Allen - FGD WWTS (Bi-Monthly Sampling) (LIMS #J13040282)

Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for total mercury and selenium speciation analysis on April 25, 2013. The samples were received in a sealed cooler at -0.5°C on April 26, 2013. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Mercury quantitation was performed via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads
Vice President
Applied Speciation and Consulting, LLC

Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Allen - FGD WWTS (Bi-Monthly Sampling) (LIMS #J13040282)

May 6, 2013

## 1. Sample Reception

Three (3) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on April 25, 2013. Three (3) additional samples in 40ml borosilicate glass bottles (provided by Applied Speciation and Consulting) were submitted for total mercury quantitation. All samples were received on April 26, 2013 in a sealed container at -0.5°C.

All samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. The 40mL borosilicate glass vials submitted for total mercury were preserved with bromine monochloride (BrCl) solution. The resulting samples were stored in a secure polyethylene container, known to be free from trace metals contamination, until the analyses could be performed.

An aliquot of each sample requiring selenium speciation evaluation was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

## 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

*Total Mercury Quantitation by CV-ICP-MS* All samples and preparation blanks for total mercury quantitation were preserved with 2% (v/v) BrCl. The resulting samples were analyzed for mercury via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS).

*Selenium Speciation Analysis by IC-ICP-CRC-MS* Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

## 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

*Total Mercury Quantitation by CV-ICP-MS* The sample fractions for total mercury quantitation were analyzed by cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS) on May 3, 2013. Aliquots of each sample are reacted with a reductant in-line and transported to a gas-liquid separator. The volatile elemental mercury that is formed is then swept by a stream of argon gas into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and separated on the basis of their mass-to-charge ratio (m/z) by a mass spectrometer. A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

*Selenium Speciation Analysis by IC-ICP-CRC-MS* Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on April 27, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

## 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

The eMDL for mercury has been calculated using the standard deviation of the preparation blanks preserved and analyzed concurrently with the submitted samples.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads
Vice President
Applied Speciation and Consulting, LLC

Total Mercury & Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD WWTS (Bi-Monthly Sampling)
Contact: Jay Perkins
LIMS #J13040282

Date: May 6, 2013
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

***Sample Results***

| Sample ID | Total Hg | Se(IV) | Se(VI) | SeCN | MeSe(IV) | SeMe | Unknown Se Species (n) |
|---|---|---|---|---|---|---|---|
| FGD Purge Eff | NR | 172 | 737 | ND (< 1.4) | 3.9 | 1.4 | 2.7 (2) |
| BioReactor 1 Inf | 0.346 | 62.7 | 111 | ND (< 0.35) | 7.27 | ND (< 0.29) | 4.93 (1) |
| BioReactor 2 Inf | 0.0718 | NR | NR | NR | NR | NR | NR |
| BioReactor 2 Eff | 0.0137 | 2.19 | ND (< 0.22) | ND (< 0.35) | ND (< 0.29) | 0.31 | 0.0 (0) |

All results reflect the applied dilution and are reported in µg/L
NR = Analysis not requested
ND = Not detected at the applied dilution
SeCN = Selenocyanate
MeSe(IV) = Methylseleninic acid
SeMe = Selenomethionine
Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS
n = number of unknown Se species observed

Total Mercury & Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD WWTS (Bi-Monthly Sampling)
Contact: Jay Perkins
LIMS #J13040282

Date: May 6, 2013
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

***Quality Control Summary - Preparation Blank Summary***

| Analyte (µg/L) | PBW1 | PBW2 | PBW3 | PBW4 | Mean | StdDev | eMDL* | eMDL 5x | eMDL 250x | eMDL 1000x |
|---|---|---|---|---|---|---|---|---|---|---|
| Hg | 0.0006 | -0.0003 | 0.0007 | 0.0000 | 0.0003 | 0.0005 | 0.0003 | 0.0014 | - | - |
| Se(IV) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.001 | - | 0.30 | 1.2 |
| Se(VI) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.001 | - | 0.22 | 0.90 |
| SeCN | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.001 | - | 0.35 | 1.4 |
| MeSe(IV) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.001 | - | 0.29 | 1.2 |
| SeMe | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.001 | - | 0.29 | 1.2 |

eMDL = Estimated Method Detection Limit
*Please see narrative regarding eMDL calculations

***Quality Control Summary - Certified Reference Materials***

| Analyte (µg/L) | CRM | True Value | Result | Recovery |
|---|---|---|---|---|
| Hg | NIST 1641d | 1568 | 1588 | 101.3 |
| Se(IV) | LCS | 4.79 | 4.819 | 100.7 |
| Se(VI) | LCS | 4.74 | 4.574 | 96.5 |
| SeCN | LCS | 4.46 | 4.496 | 100.8 |
| MeSe(IV) | LCS | 3.24 | 3.134 | 96.9 |
| SeMe | LCS | 4.66 | 4.439 | 95.3 |

Total Mercury & Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD WWTS (Bi-Monthly Sampling)
Contact: Jay Perkins
LIMS #J13040282

Date: May 6, 2013
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

***Quality Control Summary - Matrix Duplicates***

| Analyte (µg/L) | Sample ID | Rep 1 | Rep 2 | Mean | RPD |
|---|---|---|---|---|---|
| Hg | BioReactor 2 Eff | 0.0137 | 0.0139 | 0.0138 | 1.7 |
| Se(IV) | Batch QC | ND (< 0.30) | ND (< 0.30) | NC | NC |
| Se(VI) | Batch QC | ND (< 0.22) | ND (< 0.22) | NC | NC |
| SeCN | Batch QC | ND (< 0.35) | ND (< 0.35) | NC | NC |
| MeSe(IV) | Batch QC | ND (< 0.29) | ND (< 0.29) | NC | NC |
| SeMe | Batch QC | ND (< 0.29) | ND (< 0.29) | NC | NC |

ND = Not detected at the applied dilution
NC = Value was not calculated due to one or more concentrations below the eMDL

***Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate***

| Analyte (µg/L) | Sample ID | Spike Conc | MS Result | Recovery | Spike Conc | MSD Result | Recovery | RPD |
|---|---|---|---|---|---|---|---|---|
| Hg | BioReactor 2 Eff | 2.000 | 1.963 | 97.5 | 2.000 | 1.978 | 98.2 | 0.8 |
| Se(IV) | Batch QC | 1390 | 1501 | 108.0 | 1390 | 1503 | 108.1 | 0.1 |
| Se(VI) | Batch QC | 1261 | 1259 | 99.8 | 1261 | 1254 | 99.4 | 0.4 |
| SeCN | Batch QC | 1144 | 1040 | 90.9 | 1144 | 1046 | 91.4 | 0.6 |

# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

DUKE ENERGY®

Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd
Huntersville, N. C. 28078
(704) 875-5245
Fax: (704) 875-4349

Analytical Laboratory Use Only

LIMS # J13040282 | MATRIX OTHER | Samples Originating From NC___ SC___

Logged By cpk | Date & Time 4-25-13 0728

Vendor ASC | Cooler Temp (C) <6

SAMPLE PROGRAM Ground Water ___ NPDES ___ Drinking Water ___ UST ___ RCRA Waste ___

[19]Page 1 of 2
DISTRIBUTION
ORIGINAL to LAB, COPY to CLIENT

| | | |
|---|---|---|
| 1)Project Name: Allen - FGD WWTS (Bi-Monthly Sampling) | | 2)Phone No: |
| 2) Client: Don Scruggs, Robbin Jolly, Bill Kennedy | | 4)Fax No: |
| 5)Business Unit: 20003 | 6)Process: BMCEFGD | Mail Code: |
| 8)Oper. Unit: AS00 | 9)Res. Type: | 10)Resp. Center: |

Customer must Complete

MR #

Customer to complete all appropriate non-shaded areas.

Sampling conducted: 2nd and 4th Monday

[15]Preserv.: 1=HCL 2=$H_2SO_4$ 3=$HNO_3$ 4=Ice 5=None

[16]Analyses Required

| LAB USE ONLY [11]Lab ID | Se Speciation Bottle ID | [13]Sample Description or ID | Date | Time | Signature | [17]Comp. | [18]Grab | TDS | | Br (Dionex) | Metals* + Hg 245.1 | Se, soluble (no dig.) | | Hg 200.8 (V_AS&C) | | | Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | 4 | | 4 | 3,4 | 3,4 | | 5 | | | 4 | | | | |
| 2013008359 | | FGD Purge Eff | 4-19-13 | 0715 | [signature] | 6 | | | | 1 | 1 | 1 | | | | | 1 | | | | |
| 60 | | EQ Tank Eff. | 4-19-13 | 0724 | [signature] | 4 | | | | | 1 | 1 | | | | | | | | | |
| 61 | | BioReactor 1 Inf | 4-19-13 | 0729 | [signature] | 5 | | | | | 1** | 1 | | 1 | | | 1 | | | | |
| 62 | | BioReactor 2 Inf | 4-19-13 | 0734 | [signature] | 3 | | | | | 1** | | | 1 | | | | | | | |
| 63 | | BioReactor 2 Eff | 4-19-13 | 0727 | [signature] | 6 | | 1 | | 1 | 1** | | | 1 | | | 1 | | | | |
| 64 | | Filter Blk | 4-19-13 | 0845 | [signature] | 1 | | | | | | 1 | | | | | | | | | |
| 65 | | Metals Trip Blk | 4-15 | 1415 | cpk | 2 | | | | | 1** | | | | | | | | | | |
| | | | | | | Filtering of soluble Se performed in the field | | | | | | | | | | | | | | | |
| | | | | | | | | 1 | | 2 | 6 | 4 | | 3 | | | 3 | | | | |

Customer to complete appropriate columns to right.

Customer to sign & date below - fill out from left to right.

| | | | |
|---|---|---|---|
| 1) Relinquished By | Date/Time | 2) Accepted By cpk | Date/Time 4-25-13 |
| 3) Relinquished By | Date/Time | 4) Accepted By Nancy Cullivan | Date/Time Temp: -0.5 4/26/13 9:15 |
| 5)Relinquished By | Date/Time | 6)Accepted By | Date/Time |
| 7)Relinquished By cpk | Date/Time 4-25-13 | 8)Accepted By | Date/Time |
| 9)Seal/Locked By cpk | Date/Time 4-25-13 | 10) Seal/Lock Opened By | Date/Time |
| 11)Seal/Locked By | Date/Time | 12)Seal/Lock Opened By | Date/Time |

Comments

* Metals=As, Cd, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS, B by TRM/ICP 1**=No Hg analyzed

Customer, IMPORTANT! Please indicate desired turnaround.

[22]Requested Turnaround

14 Days ___X___
*7 Days ___
*48 Hr ___
*Other ___
* Add. Cost Will Apply

5-9-13

# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

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DISTRIBUTION
ORIGINAL to LAB,
COPY to CLIENT

**Analytical Laboratory Use Only**

LIMS # J13040282 | MATRIX OTHER | Samples Originating From NC___ SC___

Logged By cpk | Date & Time 4-25-13 0728 | SAMPLE PROGRAM Ground Water ___ NPDES ___ Drinking Water ___ UST ___ RCRA Waste ___

Vendor ASC | Cooler Temp (C) <6

Customer must Complete

| | | |
|---|---|---|
| 1)Project Name: Allen - FGD WWTS (Bi-Monthly Sampling) | | 2)Phone No: |
| 2) Client: Don Scruggs, Robbin Jolly, Bill Kennedy | | 4)Fax No: |
| 5)Business Unit: 20003 | 6)Process: BMCEFGD | Mail Code: |
| 8)Oper. Unit: AS00 | 9)Res. Type: | 10)Resp. Center: |

MR #

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Sampling conducted: 2nd and 4th Monday

[15]Preserv.: 1=HCL 2=$H_2SO_4$ 3=$HNO_3$ 4=Ice 5=None

[16]Analyses Required

| LAB USE ONLY [11]Lab ID | Se Speciation Bottle ID | [13]Sample Description or ID | Date | Time | Signature | [17]Comp. | [18]Grab | TDS | | Br (Dionex) | Metals* + Hg 245.1 | Se, soluble (no dig.) | | Hg 200.8 (V_AS&C) | | | Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | 4 | | 4 | 3,4 | 3,4 | | 5 | | | 4 | | | | |
| 2013008359 | | FGD Purge Eff | 4-19-13 | 0715 | [signature] | 6 | | | | 1 | 1 | 1 | | | | | 1 | | | | |
| 60 | | EQ Tank Eff. | 4-19-13 | 0724 | [signature] | 4 | | | | | 1 | 1 | | | | | | | | | |
| 61 | | BioReactor 1 Inf | 4-19-13 | 0727 | [signature] | 5 | | | | | 1** | 1 | | 1 | | | 1 | | | | |
| 62 | | BioReactor 2 Inf | 4-19-13 | 0734 | [signature] | 3 | | | | | 1** | | | 1 | | | | | | | |
| 63 | | BioReactor 2 Eff | 4-19-13 | 0727 | [signature] | 6 | | 1 | | 1 | 1** | | | 1 | | | 1 | | | | |
| 64 | | Filter Blk | 4-19-13 | 0845 | [signature] | 1 | | | | | | 1 | | | | | | | | | |
| 65 | | Metals Trip Blk | 4-15 | 1415 | cpk not | 2 | | | | | 1** | | | | | | | | | | |
| | | | | | | Filtering of soluble Se performed in the field | | | | | | | | | | | | | | | |
| | | | | | | | | 1 | | 2 | 6 | 4 | | 3 | | | 3 | | | | |

Customer to complete appropriate columns to right

Customer to sign & date below - fill out from left to right.

| | | | |
|---|---|---|---|
| 1) Relinquished By | Date/Time | 2) Accepted By cpk | Date/Time 4-25-13 |
| 3) Relinquished By | Date/Time | 4) Accepted By | Date/Time |
| 5)Relinquished By | Date/Time | 6)Accepted By: | Date/Time |
| 7)Relinquished By cpk | Date/Time 4-25-13 | 8)Accepted By: | Date/Time |
| 9)Seal/Locked By cpk | Date/Time 4-25-13 | 10) Seal/Lock Opened By | Date/Time |
| 11)Seal/Locked By | Date/Time | 12)Seal/Lock Opened By | Date/Time |

Comments

* Metals=As, Cd, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS, B by TRM/ICP 1**=No Hg analyzed

Customer, IMPORTANT! Please indicate desired turnaround.

[22]Requested Turnaround

14 Days ____X____
*7 Days _____
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5-9-13