# 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:** **J10090180**

Customer Name(s): Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy

Customer Address: 253 Plant Allen Rd.
Mail Code: ASS01
Belmont,NC 28012

Lab Contact: Jason C Perkins Phone: 704-875-5348

**Report Authorized By: (Signature)** Digitally signed by JCP7132 (111773) DN: dc=com, dc=duke-energy, dc=ent, dc=nam, ou=Accounts, ou=Personal, ou=PNTransitional, cn=JCP7132 (111773) Date: 2010.10.21 16:15:12 -04'00' **Date:** 10/21/2010

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**Program Comments:**

Allen FGD Test Burn #1

Sampled: 10/7

**Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with an "X" or "1" indicate a deviation from the method quality system or quality control requirement. All results are reported on a dry weight basis unless otherwise 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 |
|---|---|---|---|---|
| 2010008544 | ALLEN | 07-Oct-10 10:10 AM | Chris Williams | FGD Purge Eff |
| 2010008545 | ALLEN | 07-Oct-10 2:12 PM | Chris Williams | BioReactor 1 Inf |
| 2010008546 | ALLEN | 07-Oct-10 2:08 PM | Chris Williams | BioReactor 2 Eff |
| 2010008547 | ALLEN | 30-Sep-10 10:30 AM | L. Davis | Metals Trip Blank |

4 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 |
| The Vendor Laboratories have been qualified by the Analytical Laboratory | Yes | |

## 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
- ☐ Test Case Narratives
- ☑ Chain of Custody
- ☐ Electronic Data Deliverable (EDD) Sent Separately

Reviewed By: Jenny A Herman Date: 10/21/2010

# Certificate of Laboratory Analysis

*This report shall not be reproduced, except in full.*

**Order # J10090180**

Site: FGD Purge Eff — **Sample #: 2010008544**

Collection Date: 07-Oct-10 10:10 AM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|
| **SELENIUM SPECIATION** | | | | | | | |
| Vendor Parameter | Complete | | | | V_AS&C | | |

Site: BioReactor 1 Inf — **Sample #: 2010008545**

Collection Date: 07-Oct-10 2:12 PM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|
| **SELENIUM SPECIATION** | | | | | | | |
| Vendor Parameter | Complete | | | | V_AS&C | | |

Site: BioReactor 2 Eff — **Sample #: 2010008546**

Collection Date: 07-Oct-10 2:08 PM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|
| **SELENIUM SPECIATION** | | | | | | | |
| Vendor Parameter | Complete | | | | V_AS&C | | |

Site: Metals Trip Blank — **Sample #: 2010008547**

Collection Date: 30-Sep-10 10:30 AM — Matrix: OTHER

| Analyte | Result | Units | Qualifiers | RDL | Method | Analysis Date/Time | Analyst |
|---|---|---|---|---|---|---|---|
| **SELENIUM SPECIATION** | | | | | | | |
| Vendor Parameter | Complete | | | | V_AS&C | | |

APPLIED SPECIATION
AND CONSULTING, LLC

18804 Northcreek Parkway Bothell, WA, 98011
Tel: (425) 483-3300 Fax: (425) 483-9818
www.appliedspeciation.com

October 17, 2010

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

Project: Allen – FGD Alternate Fuels Test Burn #1 - Se (LIMS # J10090180)

Dear Mr. Perkins,

Attached is the report associated with three (3) aqueous samples and one associated blank submitted for selenium speciation analysis on October 11, 2010. The samples were received on October 12, 2010 in a sealed cooler at -0.5$^{o}$C. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any analytical 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 Alternate Fuels Test Burn #1 - Se (LIMS # J10090180)

October 17, 2010

## 1. Sample Reception

Three (3) aqueous samples and one associated blank in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on October 11, 2010. The samples were received on October 12, 2010 in a sealed container at -0.5$^{o}$C.

The samples were received in a laminar flow clean hood void of trace metals contamination and ultra-violet radiation. Upon reception, the samples were designated discrete sample identifiers. An aliquot of each sample was filtered (0.45µm) and these filtrates were stored in a secure, monitored cryofreezer (maintained at a temperature of -80$^{o}$C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-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.

*Selenium Speciation Analysis by IC-ICP-DRC-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 the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

## 3. Sample Analysis

All sample analysis is precluded 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 minimal interval of every ten analytical runs.

*Selenium Speciation Analysis by IC-ICP-DRC-MS* All samples for selenium speciation analysis were analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on October 14, 2010. 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 (DRC) containing a specific reactive 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 very well and no 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.

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

Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD Alternate Fuels Test Burn #1 - Se
Contact: Jay Perkins
LIMS #J10090180

Date: October 17, 2010
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

***Sample Results***

| Sample ID | Se(IV) | Se(VI) | SeCN | MeSe(IV) | SeMe | Unknown Se Species (n) |
|---|---|---|---|---|---|---|
| FGD Purge Eff | 18.8 | 448 | ND (<3.0) | ND (<2.4) | ND (<2.4) | 0 (0) |
| BioReactor 1 Inf | 5.80 | 696 | ND (<0.75) | ND (<0.60) | ND (<0.60) | 0 (0) |
| BioReactor 2 Eff | 1.23 | ND (<0.42) | ND (<0.75) | ND (<0.60) | ND (<0.60) | 0 (0) |
| Metals Trip Blk | ND (<0.13) | ND (<0.084) | ND (<0.15) | ND (<0.12) | ND (<0.12) | 0 (0) |

All results reflect the applied dilution and are reported in µg/L
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

Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD Alternate Fuels Test Burn #1 - Se
Contact: Jay Perkins
LIMS #J10090180

Date: October 17, 2010
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***Quality Control Summary - Preparation Blank Summary***

| Analyte (µg/L) | PBW1 | PBW2 | PBW3 | PBW4 | Mean | StdDev | eMDL* | eMDL 10x | eMDL 50x | eMDL 200x |
|---|---|---|---|---|---|---|---|---|---|---|
| Se(IV) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.013 | 0.13 | 0.63 | 2.5 |
| Se(VI) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.008 | 0.084 | 0.42 | 1.7 |
| SeCN | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.015 | 0.15 | 0.75 | 3.0 |
| MeSe(IV) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.012 | 0.12 | 0.60 | 2.4 |
| SeMe | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.012 | 0.12 | 0.60 | 2.4 |

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 |
|---|---|---|---|---|
| Se(IV) | ICV | 9.57 | 9.73 | 101.7 |
| Se(VI) | ICV | 9.48 | 9.23 | 97.3 |
| SeCN | ICV | 8.92 | 9.45 | 105.9 |
| MeSe(IV) | ICV | 6.47 | 6.24 | 96.4 |
| SeMe | ICV | 9.32 | 10.01 | 107.4 |

Selenium Speciation Results for Duke Energy
Project Name: Allen - FGD Alternate Fuels Test Burn #1 - Se
Contact: Jay Perkins
LIMS #J10090180

Date: October 17, 2010
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 |
|---|---|---|---|---|---|
| Se(IV) | Batch QC | 0.72 | 0.98 | 0.849 | 31.5* |
| Se(VI) | Batch QC | ND (<0.42) | ND (<0.42) | NC | NC |
| SeCN | Batch QC | ND (<0.75) | ND (<0.75) | NC | NC |
| MeSe(IV) | Batch QC | ND (<0.60) | ND (<0.60) | NC | NC |
| SeMe | Batch QC | ND (<0.60) | ND (<0.60) | NC | NC |

ND = Not detected at the applied dilution
NC = Value was not calculated due to one or more concentrations below the eMDL
*Sample concnetration is within 10x 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 |
|---|---|---|---|---|---|---|---|---|
| Se(IV) | Batch QC | 278.0 | 295.9 | 106.5 | 278.0 | 297.6 | 107.0 | 0.6 |
| Se(VI) | Batch QC | 252.3 | 253.7 | 100.6 | 252.3 | 251.0 | 99.5 | 1.0 |
| SeCN | Batch QC | 228.8 | 198.3 | 86.7 | 228.8 | 197.2 | 86.2 | 0.6 |

# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

3

**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**

Order #: J10090180
Matrix: OTHER
Samples Originating From: NC___ SC___
Logged By: J. Herman
Date & Time: 9/28/10 17:09
0846 10-8-10
Vendor: AS&C
PO# ISW01.1894
0.8 Cooler Temp (C)
SAMPLE PROGRAM: Ground Water ___ NPDES ___ Drinking Water ___ UST ___ RCRA Waste ___

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

Customer must Complete

1)Project Name: Allen - FGD
Alternative Fuels Test Burn # 1 - Se
2)Phone No:
2) Client: Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy
4)Fax No:
5)Business Unit:
6)Process:
Mail Code:
8)Oper. Unit:
9)Res. Type:
10)Resp. Center:

MR #

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

*Customer to complete all appropriate non-shaded areas.*

Sampling conducted: 2nd and 4th Monday

LAB USE ONLY

| [11]Lab ID | Se Speciation Bottle ID | [13]Sample Description or ID | Date | Time | Signature | [16]Analyses Required | [17]Comp. | [18]Grab | | |

# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

3

**Duke Energy**

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**Analytical Laboratory Use Only**

Order #: J10090180
Matrix: OTHER
Samples Originating From: NC___ SC___
Logged By: J. Herman
Date & Time: 9/28/10 17:09
Vendor: AS&C
PO# ISW01.1894
Cooler Temp (C): 0.8
MR #:

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

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

Customer must Complete

| | | |
|---|---|---|
| 1)Project Name: Allen - FGD<br>Alternative Fuels Test Burn # 1 - Se | | 2)Phone No: |
| 2) Client: Don Scruggs, Robbin Jolly, Ray Lidke, Bill Kennedy | | 4)Fax No: |
| 5)Business Unit: | 6)Process: | Mail Code: |
| 8)Oper. Unit: | 9)Res. Type: | 10)Resp. Center: |

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

*Customer to complete all appropriate non-shaded areas.*

Sampling conducted: 2nd and 4th Monday

| LAB USE ONLY [11]Lab ID | Se Speciation Bottle ID | [13]Sample Description or ID | Date | Time | Signature | [16]Analyses Required | [17]Comp. | [18]Grab | Se, speciation - vendor to [illegible] (Important to place filled bottle back into both baggies) |
|---|---|---|---|---|---|---|---|---|---|
| 2010008544 | B05810 | FGD Purge Eff | 10/10 | 1010 | Chris W Williams | | | | 1 |
| 2010008545 | B05958 | BioReactor 1 Inf | 10/10 | 1412 | Chris W Williams | | | | 1 |
| | | | | | | | | | |
| 2010008546 | B06969 | BioReactor 2 Eff | 10/10 | 1409 | Chris W Williams | | | | 1 |
| 2010008547 | B06955 | Metals Trip Blk | 4/30 | 1030 | L. Harris | | | | 1 |

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

| | |
|---|---|
| 1) Relinquished By: Chris W Williams — Date/Time: 10/7/10 1625 | 2) Accepted By: C Turner — Date/Time: 10/7/10 |
| 3) Relinquished By: C Turner — Date/Time: 10/8/10 | 4) Accepted By: L. Harris — Date/Time: 10/8/10 0745 |
| 5)Relinquished By: [illegible] — Date/Time: 10-11-10 9:00 | 6)Accepted By: — Date/Time: |
| 7)Relinquished By: — Date/Time: | 8)Accepted By: — Date/Time: |
| 9)Seal/Locked By: [illegible] — Date/Time: 10-11-10 8:30 | 10) Seal/Lock Opened By — Date/Time: |
| 11)Seal/Locked By — Date/Time: | 12)Seal/Lock Opened By — Date/Time: |

Comments: * Metals=As, Ag, B, Cu, Cr, Ni, Se, Zn

Customer, IMPORTANT: Please indicate desired turnaround

[22]Requested Turnaround

10-21-10