



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

September 8, 2011

Reply to: Donald M. Brown
Attn of: OEA-095

MEMORANDUM

Subject: Data Validation Report for the Isotope Analyses of the Samples Collected from the Yakima Basin Nitrate Study Phase 3

From: Donald M. Brown, QA Chemist^{Dmb}
USEPA Region 10, Office of Environmental Assessment, Environmental Services Unit

To: Gina Grepo-Grove, Regional Quality Assurance Manager
USEPA Region 10, OEA

The quality assurance (QA) review of the analytical data generated from the analysis of fifty-three (53) samples collected from the Yakima Basin Nitrate Study Phase 3 has been completed. All samples were analyzed for the oxygen (^{18}O) isotope of nitrate and all samples except the soil/solid samples were analyzed for the nitrogen isotope (^{15}N) of nitrate and ammonium. Sample analyses were conducted by the University of Nebraska Water Sciences Laboratory (UNL) located in Lincoln, Nebraska using in-house Standard Operating Procedures (SOPs).

This review was conducted for the following samples from the Phase 3 Study:

Well Water Samples

10154201	10154202	10154203	10154204	10154205	10154206
10154207	10154208	10154209	10154210	10154211	10154212
10154213	10154214	10154215	10154216	10154217	10154218
10154219	10154220	10154221	10154222	10154223	10154224
10154225	10154226	10154227	10154228	10154229	

Lagoon Samples

10154251	10154252	10154253	10154254	10154255	10154256
10154257	10154258	10154259	10164260	10164261	10164262
10164263	10164264	10164265			

Sewer Plant Samples

10154271	10154272	10154273			
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Soil/Solid Samples

10154245	10154246	10164237	10164238	10164239	10164240
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All sample analyses were evaluated following EPA's Stage 2B Data Validation Manual Process (S2VM). The validation was conducted according to the Quality Control Specifications outlined in the Quality Assurance Project Plan for the Yakima Basin Nitrate Study Phase 3 (April 2010) as well as the UNL Nitrogen Isotope Quality Assurance Plan and the UNL SOPs specified below. The conclusions presented herein are based solely on the information provided for this review (i.e. Quality Assurance Report and Analytical Reports).

Holding Time

Since samples were stored frozen after receipt at the laboratory, holding times were met for all samples.

Sample Results & Reporting Limits

All sample concentrations of nitrogen in nitrate and nitrogen in ammonium for the ^{15}N analysis that fell below the reporting limit (0.1 mg/L) were assigned the value of the reporting limit with the "<" symbol attached. All isotope measurements that had insufficient levels of nitrogen or oxygen were reported by the laboratory as "NM", or not measured.

Quality Control Results Summary

^{15}N Isotope Analysis of Nitrate and Ammonium

Forty-seven (47) samples were distilled and prepared for analysis following UNL SOPs *Distillation and Determination of Ammonium and Nitrate Nitrogen in Water for Nitrogen Isotope Analysis* (SOP# Analyte-DISTN15-004) and *Converting Ammonium Sulfate ((NH₄)₂SO₄) to Nitrogen (N₂) Gas on the Delta N-15 Prep Line* (SOP# Analyte-PREPN15-002). These samples were then analyzed for ^{15}N isotope in nitrate and ammonium following UNL SOP *N15 Analysis on Optima Dual Inlet IRMS* (SOP# Inst-Optima-N15-002). The following is a summary of the quality control indicators associated with the analysis, the relevant evaluation criteria, and an indication of compliance:

Laboratory Fortified Blank (LFB): The laboratory analyzed one (1) LFB with each sample batch fulfilling the frequency requirement. Recoveries were within the laboratory established control limits (Control Limits = Average \pm 3 * Standard Deviation).

Laboratory Reagent Blank (LRB): The laboratory analyzed one (1) LRB with each sample batch fulfilling the frequency requirement. The blank results were all below the reporting limit.

Laboratory Duplicate: Samples 10154201, 10154217, and 10164261 were analyzed in duplicate fulfilling the 5% frequency requirement. The duplicate results were within the laboratory established control limits (Upper Control Limit = 3.27 * Average Range).

^{18}O Isotope Analysis of Nitrate

Fifty-three (53) samples were prepared for analysis following UNL SOP *^{18}O in Nitrates by Conversion to AgNO₃* (SOP# Analyte- ^{18}O in Nitrate/AgNO₃-001). These samples were then analyzed for ^{18}O isotope in nitrate following UNL SOP *^{18}O (and ^{15}N) in Nitrates and Phosphates: Isoprime EA* (SOP# Inst-Isoprime EA- ^{18}O -001). The following is a summary of the quality control indicators associated

with the analysis, the relevant evaluation criteria, and an indication of compliance:

LFB: The laboratory analyzed one (1) LFB with each sample batch except for batch W10114, which fulfills the frequency requirement (5%) established by the QAPP for this site. The LFB results were comparable to the accepted standard value of 25.7 per mil relative to standard mean of ocean water (SMOW).

LRB: According to the SOPs associated with this analysis, LRBs are required to be performed at the beginning of the analysis. The QAPP for this site requires LRBs to be analyzed at a frequency of at least 5%. The laboratory failed to report the results of any blanks for this analysis.

Laboratory Duplicate: Samples 10154205, 10154217, and 10154221 were analyzed in duplicate fulfilling the 5% frequency requirement. The duplicate results were within the laboratory established control limits (Upper Control Limit = 3.27 * Average Range).

Data Qualifiers

The following is a list of validation qualifiers applied to the sample result(s) when needed to indicate associated out-of-control QA/QC results.

Data Qualifiers	
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.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in this sample.
JN	There is evidence that the analyte is present. The associated numerical result is an estimate.