



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

November 29, 2011

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

MEMORANDUM

Subject: Data Validation Report for the Total Nitrogen Analyses of the Soil 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: Ginna Grepo-Grove, Regional Quality Assurance Manager
USEPA Region 10, OEA

The quality assurance (QA) review of the analytical data generated from the analysis of five (5) biosolid samples collected from the Yakima Basin Nitrate Study Phase 3 has been completed. These samples were analyzed for Total Nitrogen/Solid, Ammonia Solid and Nitrate-N/Total Solid in accordance with Method AOAC 993.13, Standard Method 4500-NH₃ and Standard Method 4500-NO₃ E, respectively, by Cascade Analytical, Inc. located in Wenatchee, Washington.

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

10154231 10154233 10154235 10164237 10164239

In addition, this QA review also includes the analytical data generated from the analysis of ten (10) soil samples collected from the Yakima Basin Nitrate Study Phase 3. These samples were analyzed for Total Nitrogen/Solid, Ammonium-N and Nitrate-N/Nitrite in accordance with Method AOAC 993.13, Standard Method 4500-NH₄ H and Standard Method 4500-NO₃ F, respectively, by Cascade Analytical, Inc. located in Wenatchee, Washington.

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

10154232 10154234 10154236 10154241 10154242 10154243
10154244 10154245 10154246 10164238 10164240

All sample analyses were evaluated following EPA's Stage 4 Data Validation Manual Process (S4VM). 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). The conclusions presented herein are based on the information provided for the review.

Holding Time

In terms of holding times for nitrate and/or nitrite, the Standard Methods listed by the laboratory allow a 48-hour holding time for unpreserved, aqueous samples. The equivalent EPA Method 353.2 allows for a 28-day holding time for nitrate + nitrite and a 48-hour holding time when analyzed singly. However, all of these methods do not specify holding times for soil samples. The QAPP for this site listed a holding time of 14 days for extractable nitrate in soil samples. The laboratory analyzed the soils samples from 2 days to 8 days for these parameters.

Quality Control Results Summary

The assessment of instrument specific quality control results included instrument calibration, verification standards and blanks. Sample quality control results were assessed for matrix spike recoveries and laboratory duplicate results. The following table is a list of these quality control indicators, the relevant evaluation criteria and an indication of compliance.

Quality Control Test	Outliers?	Evaluation Criteria
Calibration and Laboratory Blanks	N	± MDL or sample <10X Blank Value
Quality Control Samples	N	85 – 115%
Matrix Spike	N	80 – 120 %
Laboratory Duplicate	N	<20% RPD

A comparison of the reported analyte values was conducted against the instrument data and the results were verified. For the purposes of this review no data were qualified.