UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 LABORATORY
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QUALITY ASSURANCE MEMORANDUM FOR ORGANIC CHEMICAL ANALYSES

DATE: February 28, 2011

TO: Curt Black, Project Manager
Office of Environmental Assessment, Risk Evaluation Unit, US EPA Region 10

From: Steven Reimer, Chemist
Office of Environmental Assessment, US EPA Region 10 Laboratory

SUBJECT: Quality Assurance Review of Yakima Nitrate Study Phase 3 Pesticides

Project Code: ESD-163C
Account Code: 1011B10P201B53C

The following is a quality assurance review of the results for pesticide analysis of sixteen soil samples. These samples were submitted for the Yakima Nitrate Study Project. The analyses were performed by EPA chemists at the US EPA Region 10 Laboratory in Port Orchard, WA, following US EPA and Laboratory guidelines.

This review was conducted for the following samples:

10154231	10154232	10154233	10154234	10154235	10154236	10154241
10154242	10154243	10154244	10154245	10154246	10164237	10164238
10164239	10164240					

Data Qualifications

Comments below refer to the quality control specifications outlined in the Laboratory's current Quality Assurance Manual, Standard Operating Procedures (SOPs) and the Quality Assurance Project Plan (QAPP).

The quality control measures which did not meet Laboratory/QAPP criteria are annotated in the title of each affected subsection with "*Laboratory/QAPP Criteria Not Met*".

For those tests for which the EPA Region 10 Laboratory has been accredited by the National Environmental Laboratory Accreditation Conference (NELAC), all requirements of the current NELAC Standard have been met.

1. Sample Transport and Receipt

Upon sample receipt, no conditions were noted that would negatively affect data quality of the results for pesticide analysis.

2. Sample Holding Times

The concentration of an analyte in a sample or sample extract may increase or decrease over time depending on the nature of the analyte. For this reason, holding time limits are recommended for samples. Samples were frozen upon receipt and extracted eight months after sampling. No holding time is listed in the QAPP for frozen samples. Normal lab holding time for frozen soils and sediments is one year. The extracts met holding time criterion.

3. Sample Preparation

Samples were prepared according to a method outlined in the Analysts Narrative based on EPA Method 3570. No SOP has been written for this analysis. No qualification of the data was required based on sample preparation.

4. Initial Calibration and Calibration Verification - Laboratory/QAPP Criteria Not Met

An initial calibration was acquired on January 25. All samples were analyzed on this instrument and evaluated against this calibration. A second initial calibration was acquired on a different instrument using different instrument condition, e.g. a different phase column, on January 31. Data was reported from the second analytical sequence with the first sequence data used to confirm.
The second initial calibration was confirmed with a standard from a second source. All target analytes met criterion (<30% deviation).
All calibration verification checks met the frequency criterion. Recovery criterion passed for the majority of target analytes, failures and affected samples are listed below.

CCV	Targets out (*over 120%*)	Samples affected
1	Fenhexamid, phosmet (imidan), azinphos methyl, surflan (oryzalin)	OBS1011A1, OBS1011F1-4
2	*Oxyfluorfen, propargite, surflan*	OBS1014A1, OBS1014F1-2, 10154232,10154233, 10154234, 10154235, 10154235S1-2
3	Fenhexamid, imidan, azinphos methyl,	10154236, 10154241, 10154242, 10154243, 10154244, 10154245
4	*Oxyfluorfen,* azinphos methyl, imidan, *surflan*	10154246, 10164237, 10164238, 10164239,
5	Fenhexamid, imidan, azinphos methyl, imidan, surflan	10164240, 10164240D
6	azinphos methyl, imidan,	Dilutions of 10154245, 154231

Due to the recurring failures the results for fenhexamid, phosmet (imidan), azinphos methyl and surflan (oryzalin) are qualified as estimated “J/UJ” in all samples. Diuron is only determined as a breakdown product, as such is qualified as estimated “J/UJ” in all samples.

5. Laboratory Control Samples - Laboratory/QAPP Criteria Not Met

Four laboratory control samples were created and analyzed. The results met the 70 to 130% recovery acceptance criteria for the method, with the exception of diuron, metribuzin, azinphos methyl and surflan . A series of LOQ spikes were created at a nominal concentration of twice the reporting limit. All target analytes were detected except diuron and surflan, only fenhexamid and azinphos-methyl had measurable recovery less than 50%. Diuron, azinphos methyl and surflan are qualified from above, metribuzin is also qualified as estimated “J/UJ” in all samples.

6. Blank Analysis

The method blank did not contain detectable levels of analytes.

7. Matrix Spike/Matrix Spike Duplicate Analysis - Laboratory/QAPP Criteria Not Met

Matrix spike analyses were performed on samples 10154235 and 10164240. The results met criteria, (recoveries between 50 and 200%, rpd<30%) except for surflan in 10154235.

8. Identification

The retention times for all detected target compounds were within acceptable limits of the initial calibration standards. Criteria were met for mass spectral ion matching and ion abundance matching or were judged acceptable.

9. Data Qualifiers

All requirements for data qualifiers from the preceding sections were accumulated. Each sample data summary sheet and each compound was checked for positive or negative results. From this, the overall need for data qualifiers for each analysis was determined. In cases where more than one of the preceding sections required data qualifiers, the most restrictive qualifier has been added to the data.

Below are the definitions for the codes used for qualifying data from these analyses. When more than one quality issue was involved, the most restrictive qualifier has been attached to the data.

U - The analyte was not detected at or above the reported value.

J - The identification of the analyte is acceptable; however the reported value is an estimate.

UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.

The usefulness of qualified data should be treated according to the severity of the qualifier in light of the project's data quality objectives. Should questions arise regarding the data, contact Steve Reimer at the Region 10 Laboratory, phone number (360) 871-8718.