



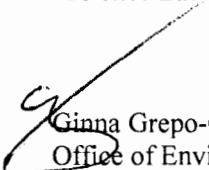
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

September 22, 2011

Reply to: OEA-095
Attn of: grepo-grove.gina@epa.gov

MEMORANDUM

Subject: QA Narrative Report for the Steroid Hormone Analysis of Samples Collected from the Phase 3 Yakima Valley Nitrates Study by University of Nebraska Lincoln – Water Science Laboratory

From:  Gina Grepo-Grove, R10 QA Manager
Office of Environmental Assessment, USEPA

To: Michael Cox, Project Officer, OEA
Curt Black, Project Manager
Sheila Fleming, Unit Manager, REU, OEA

CC: Stephen R Hutchins, TOPO, USEPA, NRMRL

The quality assurance (QA) review of the analytical data generated from the analysis of 63 samples collected from the above referenced site has been completed. The analyses were performed by the University of Nebraska Water Science Laboratory located in Lincoln, NE. The samples were analyzed for steroid hormones [11-Keto Testosterone, 17 α -hydroxyprogesterone, 17- α -estradiol (α -E2), 17- β -estradiol (β -E2), 17- α -ethynylestradiol (EE2), 17 α -trenbolone, 17 β -trenbolone, 4-androstenedione, androsterone, androstanedione, a-zearalenol a-zearalanol, b-zearalenol b-zearalanol epitestosterone, estriol, estrone, melengestrol acetate, progesterone and testosterone following University of Nebraska Water Science Laboratory (UNL) Standard Operating Procedures “*Analysis of steroids in water samples by on-line solid-phase extraction (SPE) and high pressure liquid chromatography tandem mass spectrometry (HPLC/MS/MS)*”, SOP# LCMS_APPI_STEROIDS_WATER-001 and “*Analysis of Steroids in solid samples (i.e. soils, manure, etc) by microwave-assisted solvent extraction (MASE) and liquid chromatography-tandem mass spectrometry (LC/MS/MS)*” (SOP# Analyte-Steroids_Solids-001.

The following samples were evaluated in this validation report:

Lagoon Samples:

LG01	LG02	LG03	LG04	LG05	LG06
LG07	LG08	LG09	LG10	LG11	LG12
LG13	LG14	LG15			

Waste Water Treatment Plant (WWTP) Samples

SP01 SP02 SP03

Water Samples

WW-01	WW-02	WW-03	WW-04	WW-05	WW-06
WW-07	WW-08	WW-09	WW-10	WW-11	WW-12
WW-13	WW-14	WW-15	WW-16	WW-17	WW-18
WW-19	WW-20	WW-21	WW-22	WW-23	WW-24
WW-25	WW-26	WW-27	WW-28	WW-29	

Solid Samples

SO-01	SO-02	SO-03	SO-04	SO-05	SO-06
SO-07	SO-08	SO-09	SO-10	SO-11	SO-12
SO-13	SO-14	SO-15	SO-16		

DATA QUALIFICATIONS

All sample analyses were evaluated following the EPA's Stage 2B Manual Data Validation Process (S2VM). The analyses were evaluated and laboratory qualifiers were mapped to R10 EPA validation qualifiers following the technical acceptance criteria and Quality Control Specifications outlined in the UNL Standard Operating Procedure and the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA-540-R08-005).

Limitations of the Data Validation:

The following documents were used as supporting documentation for data review:

- *QA Report UNL June 2011*
- *Summary of Sample Analytical Results*
- *Steroid and Hormones Calibration 0111811*
- *Steroid and Hormones Calibration 020411*
- *Steroid and Hormones Calibration 020811*
- *Steroid and Hormones Sequence and Internal Standard Report 2011*

The conclusions presented herein are based on the information provided for the review. None of the detected results and calculations were verified using the instrument raw data.

Overall Data Assessment:

Samples were analyzed following the technical specifications of the SOPs. The technical acceptance criteria for all analyses were met. Due to the calibration results, the detected results and/or reporting limits for androstenedione, a-methyltestosterone, androsterone, progesterone, caffeine, estrone, a-zearalanol, a-zearalenol, b-zearalanol for samples associated with the calibration run on 01/18/2011 were qualified estimated, “J/UJ”. Approximately 15% of the total data points were flagged estimated due to calibrations. In addition, target compounds that were detected in both field blank and the associated sample at concentrations <5x the value in the blank were qualified as non-detects, “U”. The data, as qualified, are usable for all purposes.

Data Qualifiers

The following is a list of validation qualifiers applied to the sample result(s) when needed to indicate an 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.