GWERD QUALITY ASSURANCE PROJECT PLAN

Title: Hydraulic Fracturing Retrospective Case Study, Wise, TX: Analysis of Samples by the EPA Region VII Contract Laboratory for the September and December 2012 Sampling Events

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EPA does not consider this internal planning document an official Agency dissemination of information under the Agency's Information Quality Guidelines, because it is not being used to formulate or support a regulation or guidance; or to represent a final Agency decision or position. This planning document describes the overall quality assurance approach that will be used during the research study. Mention of trade names or commercial products in this planning document does not constitute endorsement or recommendation for use.

The EPA Quality System and the HF Research Study

EPA requires that all data collected for the characterization of environmental processes and conditions are of the appropriate type and quality for their intended use. This is accomplished through an Agency-wide quality system for environmental data. Components of the EPA quality system can be found at http://www.epa.gov/quality/. EPA policy is based on the national consensus standard ANSI/ASQ E4-2004 *Quality Systems for Environmental Data and Technology Programs: Requirements with Guidance for Use*. This standard recommends a tiered approach that includes the development and use of Quality Management Plans (QMPs). The organizational units in EPA that generate and/or use environmental data are required to have Agency-approved QMPs. Programmatic QMPs are also written when program managers and their QA staff decide a program is of sufficient complexity to benefit from a QMP, as was done for the study of the potential impacts of hydraulic fracturing (HF) on drinking water resources. The HF QMP describes the program's organizational structure, defines and assigns quality assurance (QA) and quality control (QC) responsibilities, and describes the processes and procedures used to plan, implement and assess the effectiveness of the quality system. The HF QMP is then supported by project-specific QA project plans (QAPPs). The QAPPs provide the technical details and associated QA/QC procedures for the research projects that address questions posed by EPA about the HF water cycle and as described in the *Plan to Study the Potential Impacts of Hydraulic Fracturing on Drinking Water Resources* (EPA/600/R-11/122/November 2011/www.epa.gov/hydraulic fracturing). The results of the research projects will provide the foundation for EPA's 2014 study report.

This QAPP provides information concerning the Chemical Mixing; and Flowback and Produced Water stages of the HF water cycle as found in Figure 1 of the HF QMP and as described in the HF Study Plan. Appendix A of the HF QMP includes the links between the HF Study Plan questions and those QAPPs available at the time the HF QMP was published.

Analysis of Samples for Metals by ICP-MS and ICP-OES, Mercury by Cold Vapor Atomic Absorption Spectrometry (AAS) and Volatile Organic Compounds using GC/MS by Southwest Research Institute

Purpose

The purpose of this Addendum to the QAPP for the Hydraulic Fracturing Retrospective Case Study, Wise, TX is to provide specifications and quality control (QC) acceptance criteria for the analysis of samples collected in September 2012 for metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) and Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). Samples collected in December 2012 were also analyzed for metals by ICP-MS and ICP-OES in addition to mercury by cold vapor AAS and volatile organic compounds (VOCs) by purge and trap-GC/MS.

The samples were analyzed through a Region 7 contract with ARDL, Inc. Southwest Research Institute (SwRI) is a subcontractor to ARDL, Inc. In previous sampling events, these samples were analyzed by Shaw for metals and VOCs, and by an EPA Superfund Analytical Services Contract Laboratory for metals.

Sample Handling and Custody

Samples were packed in coolers (on ice) and shipped overnight via UPS or FedEx to the laboratory, with appropriate chain of custody forms, and the cooler was sealed with custody seals.

Sample receipt and log-in was conducted as described in SwRI SOP #TAP-01-0103-016, "Sample Receipt Inspection."

Analytical Methods

The contract laboratory analyzed water samples for Al, As, Cd, Cr, Cu, Mo, Ni, Pb, Sb, Se, Sr, Th, Tl, U, and V by ICP-MS. In addition, the contract laboratory analyzed water samples for Ag, B, Ba, Be, Ca, Co, Fe, K, Li, Mg, Mn, Mo, Na, P, S, Sb, Si, Sr, Ti, and Zn by ICP-OES. For the September 2012 sampling event, the analysis did not include Hg because the sample holding time was exceeded. The contract laboratory performed the analysis in accordance with the EPA Methods 6020A for ICP-MS and 200.7 for ICP-OES. Both total and dissolved metals were analyzed. Sample digestion for total metals was done according to EPA Method 200.7. Samples for dissolved metals were not digested. Samples collected in December 2012 were also analyzed for mercury and volatile organic compounds in accordance with EPA Methods 7470A and EPA Method 8260B, respectively.

SOW and Project Plan Specifications:

A Statement of Work (SOW) was prepared and QA-approved prior to submitting the samples to the laboratory for analysis describing requirements for analytical methods and QA/QC.

The SOW Reporting Limits (RLs) are listed in the following tables.

Analyte	ICP-AES* RL (mg/L)	ICP-MS RL (µg/L)
Al	0.200	20
Sb		2
As		0.2
Ba	0.200	
Be	0.005	
Cd		0.2
Ca	0.5	
Cr		2
Co	0.050	
Cu		2
Fe	0.100	
Pb		0.10
Mg	0.5	
Mn	0.015	
Ni		1
K	0.5	
Se		1
Ag	0.010	
Na	1.75	
Tl		0.24
V	0.050	1
Zn	0.060	

Additional Analytes	ICP-AES RL (mg/L)	ICP-MS RL (µg/L)
B	0.35	-
Li		3
Mo		1
P	0.06	-
Si	0.5	-
Sr		5
Th		1
Ti	0.01	-
U		1

*AES: Atomic Emission Spectroscopy, equivalent to OES
(Note that Reporting Limits are equivalent to Quantitation Limits. SwRI uses RL in their reporting.) SOW requirement for the Reporting Limit for Hg by CV AAS is 0.2 µg/L.

The dissolved metals for ICP-MS or-OES analysis shall not be digested. If any ICP-MS analyte is detected by ICP-OES at levels equal to or greater than 100 times the ICP-MS RLs, that analyte will be reported from the ICP-OES and not ICP-MS.

TARGET COMPOUNDS	*TARGET LIMITS (µg/L)*	
	MDL	**QL or LOQ**
1,1,1-Trichloroethane	0.13	0.5
1,1,2-Trichloroethane	0.21	0.5
1,1-Dichloroethane	0.13	0.5
1,1-Dichloroethene	0.12	0.5
1,3,5-Trimethylbenzene	0.05	0.5
1,2,4-Trimethylbenzene	0.05	0.5
1,2-Dichlorobenzene	0.1	0.5
1,2-Dichloroethane	0.21	0.5
1,2,3-Trimethylbenzene*	0.07	0.5
1,3-Dichlorobenzene	0.16	0.5
1,4-Dichlorobenzene	0.17	0.5
Acetone	3.45	10
Benzene	0.06	0.5
c-1,2-Dichloroethene	0.14	0.5
Carbon disulfide	0.21	0.5
Carbon tetrachloride	0.12	0.5
Chlorobenzene	0.08	0.5
Chloroform	0.13	0.5
Diisopropyl ether*	0.11	1
Ethanol*	18	100
Ethyl benzene	0.06	0.5
Ethyl t-butyl ether*	0.08	1
Isopropyl alcohol*	2.37	10
Isopropyl benzene	0.05	0.5
m/p-Xylene	0.09	1
Methyl t-butyl ether	0.09	1
Methylene chloride	0.21	1
Naphthalene	0.31	1
o-Xylene	0.08	0.5

t-1,2-Dichloroethene	0.1	0.5
t-Amyl methyl ether*	0.09	1
t-Butyl alcohol*	2.41	10
Tetrachloroethene	0.13	0.5
Toluene	0.08	0.5
Trichloroethene	0.09	0.5
Vinyl chloride	0.18	0.5
Acrylonitrile		25

*These compounds were not routinely analyzed previously by the laboratory and required a new MDL study.

An MS/MSD (Matrix Spike/Matrix Spike Duplicate) and LCS (Laboratory Control Sample) shall be analyzed for every 20 samples received and/or for a given site as indicated on the shipping document.

Quality Control

The following Tables 1-4 summarizes the acceptance criteria and frequency for the QC checks conducted during the course of sample analysis.

Table 1. QC Checks for ICP-MS

QC Type or Operation	Acceptance Criterion	Frequency
Instrument Calibration	The acceptance criterion for the initial calibration correlation coefficient is $r \geq 0.998$.	Daily. Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment. The lowest non-blank standard shall be set at the RL for all analytes.
Initial Calibration Verification	90-110% Recovery	Following instrument calibration for each mass used.
Initial Calibration Blank	$\leq$RL	Following each instrument calibration, immediately after the ICV.

QC Type or Operation	Acceptance Criterion	Frequency
Continuing Calibration Verification	90-110% Recovery	For each mass used, at a frequency of at least after every 10 analytical runs, and at the end of each run.
Low Level Initial Calibration Verification (LLICV) and Low Level Continuing Calibration Verification (LLCCV) at the RL (identified by lab as CRDL)	70-130% Recovery	LLICV, following each instrument calibration., and LLCCV analyzed at the end of each run.
Continuing Calibration Blank	≤RL	At a frequency of at least after every 10 analytical runs, and at the end of each run. Performed immediately after the last CCV.
Interference Check Sample	For solution AB, ±20% of the analyte's true value; for solution A ±5 ppb or ±2 times the RL of the analyte's true value, whichever is greater.	At the beginning of the run after the ICB but before the CCV.
Serial Dilution	If the analyte concentration is sufficiently high (minimally a factor of 50 above the RL in the original sample), the serial dilution (a five-fold dilution) shall then agree within 10% of the original determination after correction for dilution.	Every 20 samples.
Preparation or Method Blank	≤RL	Every 20 samples.
Laboratory Control Sample	80-120% Recovery	Every 20 samples.

QC Type or Operation	Acceptance Criterion	Frequency
Matrix Spike	75-125% Recovery (Recovery calculations are not required if sample concentration >4x spike added.)	Every 20 samples.
Post-Digestion Spike	80-120% Recovery per 6020A (Note that the lab SOP uses 75-125% Recovery)	Each time Matrix Spike Recovery is outside QC limits.
Duplicate Sample	RPD≤20% for sample values ≥5x RL	Every 20 samples.
ICP-MS Tune	Mass calibration must be within 0.1 amu of the true value in the mass regions of interest. The resolution must also be verified to be less than 0.9 amu full width at 10% peak height.	Prior to calibration.
Internal Standards	The absolute response of any one internal standard in a sample must not be <70% from the response in the calibration standard.	Internal standards shall be present in all samples, standards, and blanks (except the tuning solution) at identical levels.
Determination of Method Detection Limits		Annually and after major instrument adjustment.

Table 2. QC Checks for ICP-OES

QC Type	Acceptance Criteria	Frequency
Instrument Calibration	Criteria not given in 200.7.	Daily. Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification (QCS or Quality Control	95-105% Recovery	Immediately after calibration.

QC Type	Acceptance Criteria	Frequency
Standard)		
Initial Calibration Blank	≤RL	Analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument.
Continuing Calibration Verification (IPC or Instrument Performance Check)	90-110% Recovery	At beginning and end of run; every 10 samples during analytical run.
Continuing Calibration Blank	≤RL	Analyzed immediately after every Continuing Calibration Verification (CCV); at beginning and end of run and every 10 samples during an analytical run.
Interference Check Sample (SIC or Spectral Interference Check)	For solution AB, ±20% of the analyte's true value; for solution A ±20% of the interferent's true value, for all other analytes ±5 ppb or within ±2 times the RL of the analyte's true value, whichever is greater.	At the beginning of the run after the ICB but before the CCV and at the end of the run.
Serial Dilution	If the analyte concentration is sufficiently high (minimally a factor of 50 above the MDL in the original sample), the serial dilution (a five-fold dilution) shall then agree within 10% of the original determination after correction for dilution.	Every 20 samples.
Preparation Blank (LRB or Laboratory Reagent Blank)	≤RL	Every 20 samples.
Laboratory Control Sample (LFB or Laboratory Fortified Blank)	85-115% recovery	Every 20 samples.
Matrix Spike (LFM or Laboratory Fortified Matrix)	75-125% Recovery (Recovery calculations are not required if	Every 20 samples.

QC Type	Acceptance Criteria	Frequency
	sample concentration >4x spike added.)	
Post-Digestion Spike	85-115% Recovery	Each time Matrix Spike Recovery is outside QC limits.
Duplicate Sample	RPD≤20% for sample values ≥5x RL; for sample values <5xRL, control limit = RL	Every 20 samples.
Determination of Method Detection Limits		Annually and after major instrument adjustment.

Table 3. QC Checks for Mercury by Cold Vapor AAS

QC Type	Acceptance Criteria	Frequency
Instrument Calibration	The acceptance criterion for the initial calibration correlation coefficient is r≥0.995.	Daily. Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment. The lowest non-blank standard shall be set at the RL.
Initial Calibration Verification (ICV, second source)	90-110% Recovery	Immediately after calibration.
Initial Calibration Blank (ICB)	≤RL	Analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument.
Continuing Calibration Verification (CCV)	90-110% Recovery	Every 10 samples and at the end of the run.
Lower Limit of Quantitation Check (LLQC) (identified by lab as either CRI or CRA)	70-130% Recovery	Analyzed at beginning and the end of each run.
Continuing Calibration Blank (CCB)	≤RL	Analyzed immediately after every Continuing Calibration Verification (CCV); every 10 samples and at the end of the

QC Type	Acceptance Criteria	Frequency
		run.
Method or Preparation Blank	≤RL	Every 20 samples.
Laboratory Control Sample	80-120% recovery	Every 20 samples.
Matrix Spike	75-125% Recovery (Recovery calculations are not required if the sample concentration is >4x the spike added.)	Every 20 samples.
Post-Digestion Spike	80-120% Recovery per Method 7000B as reference in 7470A (Note the lab sop uses 75-125% Recovery)	If a MS and/or MSD are out of control.
Duplicate Sample	RPD≤20% for sample values ≥5x RL; for sample values <5xRL, control limit = RL	Every 20 samples.
Determination of Method Detection Limits		Annually and after major instrument adjustment.

Table 4. QC Checks for VOCs by GC/MS

QC Type	Acceptance Criteria	Frequency
Instrument Calibration	The acceptance criterion for the initial calibration requires RSD ≤15% or for alternate curve fits the correlation coefficient r≥0.990.	Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment. The lowest non-blank standard shall be set at the RL.
System Performance Check	BFB Tune must meet tuning criteria in Table 4 of 8260B. Minimum average response factors for the SPC compounds* must meet criteria	Prior to sample analysis; beginning of each 12 hour shift.

QC Type	Acceptance Criteria	Frequency
Initial Calibration Verification (second source)	75-125% Recovery	Immediately after calibration.
Continuing Calibration Verification (CCV)	80-120% Recovery	Every 12 hours.
Surrogates	70-130% Recovery	All blanks, QC samples, and samples.
Internal Standards	EICP area must not vary by more than a factor of 2 (-50 to +100%) of the mid-point calibration standard. Retention time must not vary by more than 0.50 min of those in the mid-point calibration standard.	All blanks, QC samples, and samples.
Method Blank	≤RL ≤2xRL for methylene chloride, acetone, and 2-butanone	After calibration standards. Every 12 hours.
Laboratory Control Sample	70-130% Recovery 60-140% Recovery for t-butyl alcohol, isopropyl alcohol, and ethanol	Every 20 samples.
Matrix Spike	70-130% Recovery 60-140% Recovery for t-butyl alcohol, isopropyl alcohol, and ethanol	Every 20 samples.
Duplicate Sample (MS/MSD)	RPD≤30%	Every 20 samples.
Determination of Method Detection Limits		Annually and after major instrument adjustment.

*SPC compounds minimum response factors (RF):

Chloromethane, min. RF = 0.10
1,1-Dichloroethane, min. RF = 0.10
Bromoform, min. RF = 0.10
1,1,2,2-Tetrachloroethane, min. RF = 0.30
Chlorobenzene, min. RF = 0.30

Data Review and Validation

The laboratory performed data review according to their SOP #TAP-01-0103-014, "Data Review and/or Data Validation."

A QA contractor or EPA staff, under the direction of the GWERD Quality Assurance Manager (QAM) will subsequently conduct an Audit of Data Quality on the data set according to NRMRL SOP LSAS-QA-02-0 "Performing Audits of Data Quality (ADQs)". The auditors will review the information presented in the data report, review the data, and ensure that appropriate project-specific data qualifiers are included in the data tables. Data transcription checks of 100% of the data will also be performed.

Reporting Requirements

Data deliverables were required in electronic format. The electronic data deliverable was to be provided to the RASP PO by 2:00pm CST on the 21st day after receipt of the last sample for a given sampling event. (NOTE: If the due date falls on a Holiday, Saturday or Sunday, then the deliverables are due to EPA by 12:00pm on the first subsequent business day). Electronic deliverables included all analytical results (field and laboratory QC samples) and the associated narrative. In addition to the normal narrative and Excel spreadsheet required, the laboratory provided an electronic "CLP type" data package that included the written narrative, Forms 1's, QC data, & all supporting raw data. The package was organized and paginated. The entire data package was provided in a .pdf file format. The complete data package in .pdf format was provided within 48 hours of the electronic results and narrative.

NOTE: The associated narrative addressed each of the applicable areas listed below for every parameter group in the task order. This included a statement that the QA/QC criteria for every applicable area were in control or, conversely, that one or more QC outliers were present. For areas with outliers, the narrative specified each parameter which was out of control and the associated samples that were affected. In addition, the narrative indicated any and all corrective actions taken and the results of those actions as well as impact on the associated samples.

- Holding Times
- Initial Calibration
- Continuing Calibration
- Surrogates
- Internal Standards
- Laboratory Duplicate
- Matrix Spike/Matrix Spike Duplicate
- Laboratory Control Sample
- Method Blanks

Revision History

Revision Number	Date Approved	Revision

0	6/20/11	New document
1	2/27/12	• Added $^{87}Sr/^{86}Sr$ isotopes and O,H stable isotopes of water to analyte list to ascertain if the water is from a different source or is mixture of aquifer water and source water. (Sections 2.2.1.1, 2.4.1, 2.5.1, and Tables 6, 8, 9, and 13) • Added USGS Laboratory contact information (2.3.3) • Added Appendix A for Sr isotope methodology used by USGS • Revised Project /Task organization (Section 1.1) to reflect change in personnel • Revised location information (1.2.4 and 1.2.5) • Updated Region VIII accreditation status and text to 2nd paragraph to provide clarification (1.5) • Added geophysical measurements and methods to help identify the source of contamination and determine the extent of contamination (2.1.3 and 2.2.2) • Added USGS sample shipping information (2.3.3) • Section 2.2.1.1, #4o, made corrections to cited methods • Section 2.5.1, for Region VIII, #5, indicated that Region VIII has provided their results for performance evaluations • Section 2.7, provided clarification of steps taken to check performance of field measurements for sulfide, ferrous iron, alkalinity, and turbidity • Sec. 3.1, provided clarification that ADQs are performed on the first data sets • Sec. 3.1.2 and 3.2, corrected to whom audit reports are submitted • Sec. 4.2, added text to clarify data verification/validation

		process as well as addition of new Table 18 on Data Qualifiers • Added references for geophysical measurements, stable isotopes, data review, low molecular weight acids, microwave digestion for unfiltered metals samples • Revised Table 2 Field Activities Schedule • Made corrections to methods in Table 5 (methods for ferrous iron and sulfide are not EPA); replaced alkalinity method # with correct #; added pH, DO, ORP, and specific conductance • Added bromide analysis by RSKSOP-288v3 in Tables 6 and 11; this method can analyze for Br in samples with high chloride concentrations • Revised Region VIII SVOC, Table 12 with updated limits • Table 13, corrections were made for DIC/DOC • Table 14, replaced with updated/corrected version from RegionVIII • Added Table 16 showing USGS QA/QC requirements for Sr isotopes • Revised Figure 1 and Sec. 1.1 to reflect current project organization (replaced Puls with Jewett; added Peterman, Costantino, Groves, and McElmurry)
2	5/25/12	• Section 1.1, added new data management duties for Susan Mravik. • Updated section 1.2.2 Phase 2 Investigations to reflect how phase 2 GW sampling will be done • Updated section 2.2.1.1 Domestic wells to reflect how sampling will occur for Phase 2 GW sampling • Updated Section 2.3.3. Replaced Shaw lab contact person due to departure of employee • Updated 2.4.1. Modified first sentence for clarification. • Updated Section 2.5.3. Added text on isotope analysis and

		detection limits • Updated Section 2.5.4. Added language describing process for evaluating field duplicates and blanks • Updated Section 3.1.1. Added text clarifying that PE samples are not available or needed for isotope analysis • Revised Table 2 Field Activities Schedule to reflect modified sampling frequency and ceasing of sampling at Locations A and C • Table 6, replaced EPA Method 200.7 with 6010C; both are ICP-MS methods, but 6010C is the more appropriate method based on SW846 inherent method flexibility • Table 13, replaced metals QC criteria with revised criteria to make them more consistent with 6010C • Revised Table 18 to clarify qualifiers and add new ones
3	9/10/12	• Title changed to reflect we are not doing any investigations in Denton Co., TX and to identify the special sampling event in September 2012 • Distribution list changed to reflect additional personnel involved with the study • Section 1.1 changed to reflect update to David Jewett's responsibilities • Section 1.2, Denton Co., TX removed, no longer part of project • Section 1.2.1, added description of this sampling event • Section 1.2.2, changed sampling date for Phase 2 from May 2012 to November-December 2012 • Section 1.2.3, deleted Ra because at this time no evidence suggests this to be important parameter in Wise Co. • Section 1.2.6, added information supplied to us by the homeowner and TRRC. • Section 1.3, added information on March 2012 sampling; added strontium and stable water isotopes to analyte list; added statement on the September 2012 sampling event • Section 1.3, deleted DRO and GRO. Data from previous

		sampling events indicated that these have limited utility for the study. • Section 2.2, added information for sampling production wells and scope of sampling for this sampling event, including samples for metals analysis by a CLP lab and samples for iodide • Section 2.3.2, added information for CLP lab • Section 2.3.3, added information for CLP lab • Section 2.4.1, changed title from "Ground Water" to "Ground, Surface, and Produced Waters" to reflect scope of samples to be analyzed; added analysis of iodide for this sampling event; added analysis of samples for metals by CLP lab • Section 2.5.1, added information for CLP lab analysis of metals • Section 4.2, added data validation of metals data from CLP lab • Table 2, added the September 2012 sampling event • Table 6; replaced EPA Method 6010C with 200.7 (ICP-OES analysis for metals). 200.7 was referenced in QAPP Revisions 0 and 1. It was changed in Revision 2 to 6010C but since then it was determined by QA staff that use of 200.7 as the "base" method was appropriate as 200.7 incorporates 6010C by reference. Added volume and bottle requirements for metals analysis by CLP lab. Added Iodide analysis to table. • Table 7, all instances of RL were replaced with QL; criteria for blanks were updated to be consistent with data qualifier table. • Table 11, replaced EPA Method 6010C with 200.7 (see above); replaced SOPs listed in table with their corresponding EPA Methods, and added footnotes to indicate the SOPs that implement these EPA Methods • Table 13, added table of CLP CRQLs for metals • Table 14, added Laboratory Control Sample and information for iodide • Table 18, added CLP lab QA/QC requirements • Table 20, table replaced with most recent version; U, U1, D, and T removed as they will not used; J10 has been added.
3,	11/30/12	• Title changed to reflect the focus of this addendum

Addendum		• Scope of addendum is limited to the CLP metals reanalysis
3, Addendum No. 2	1/10/13	• Title changed to reflect the focus of this addendum • Scope of addendum is limited to the SwRI analysis of samples for metals, mercury, and VOC analysis