

Monitoring Results of Natural Gas Wells near the Rulison, Colorado, Site September 2019 Monitoring Event

U.S. Department of Energy Office of Legacy Management Grand Junction, Colorado

Date Sampled: September 24, 2019

Background

The Rulison, Colorado, Site is in the Piceance Basin of western Colorado, 40 miles northeast of Grand Junction. The U.S. Atomic Energy Commission (a predecessor agency to the U.S. Department of Energy [DOE]), in partnership with Austral Oil Company Inc. and the nuclear engineering firm CER Geonuclear Corporation, conducted an underground nuclear test at the site on September 10, 1969. The Rulison site is identified as Lot 11 on the site map (Figure 1). The test, known as Project Rulison, was designed to evaluate the feasibility of using a nuclear detonation to fracture low-permeability gas-bearing sandstone reservoirs to improve gas production. This was the second natural gas stimulation experiment in the Plowshare Program, a program initiated to develop peaceful uses for nuclear energy. The exploratory well (R-Ex) was drilled at the site to determine the optimum depth for the test within the targeted formation. The device was detonated in the emplacement well (R-E) at a depth of 8425 feet (ft) and had a reported yield of 40 kilotons (DOE 2015). The detonation created a temporary cavity, a subsequent collapse chimney, and a fractured zone surrounding the cavity (collectively known as the detonation zone).

In October 1970, a year after the detonation, a reentry well was drilled into the upper part of the chimney to evaluate the test. The reentry well (R-En) was a sidetrack hole drilled directionally from the nearby exploratory well, which is about 300 ft northwest of the emplacement well. Production testing on the reentry well produced 455 million cubic feet (MMCF) of gas in 107 days from October 1970 through April 1971 (Reynolds 1971). Analysis of data collected during production testing indicated that essentially all tritium present as tritiated methane was removed from the detonation zone, but that tritium likely remained in the detonation zone as tritiated water (liquid and vapor) and some remained in minerals that make up the melt rock. In 1976, the participating parties agreed there would be no future gas production at the site, the reentry well was abandoned, and a deed restriction was established for Lot 11. The deed restriction prohibits penetration or withdrawal of any material below 6000 ft within the boundary of Lot 11 unless authorized by the U.S. government.

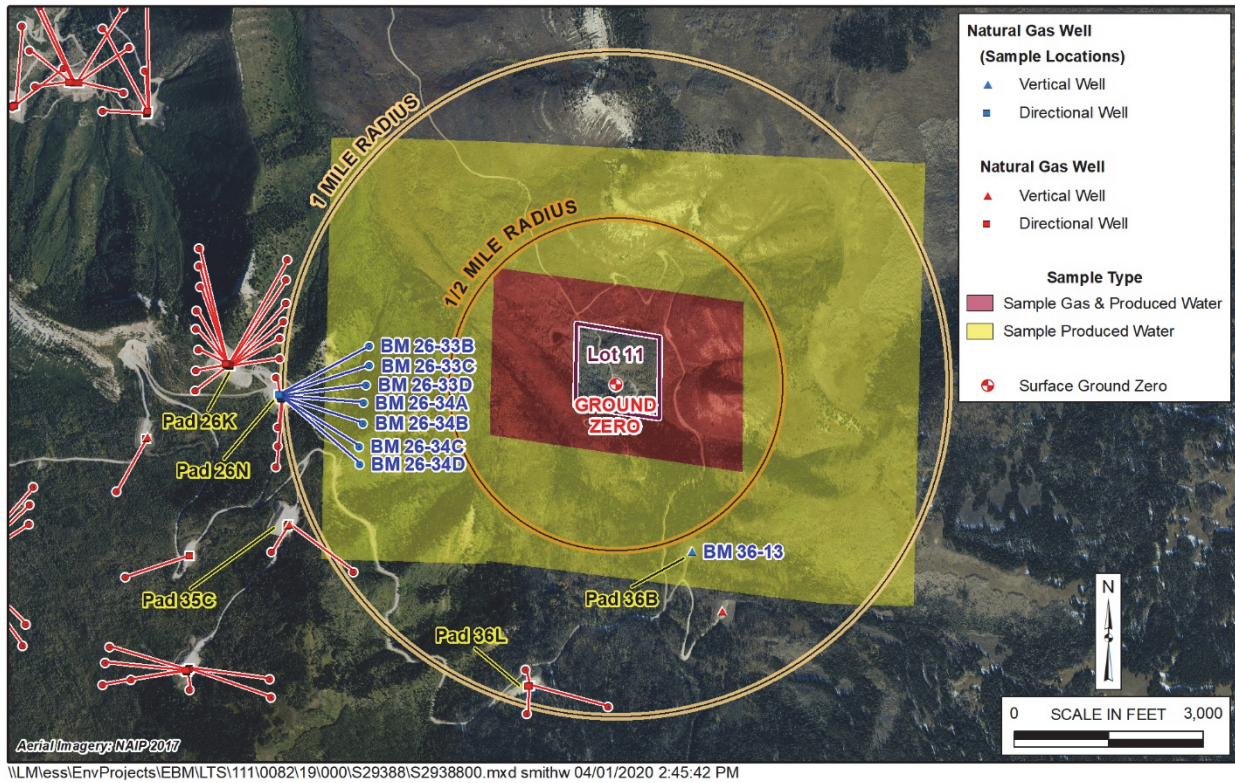


Figure 1. Rulison, Colorado, Site and Well Location Map

Purpose

DOE’s Office of Legacy Management (LM) collects samples (natural gas and produced water) from producing natural gas wells near the site to confirm no migration of detonation related radionuclides. Tritium is the most abundant radionuclide remaining in the detonation zone that can be present in both the gas and aqueous phases. This is based on estimated inventories of radionuclides produced by the detonation and the amounts removed by production testing. Tritium’s presence in water vapor (a minor constituent of natural gas) is the primary concern, because gas is more mobile than liquid in a gas reservoir. Almost all tritiated methane was removed during the production testing (Smith 1971).

Most natural gas wells produce some liquids (produced water and hydrocarbon condensate) along with natural gas; these liquids are brought to the surface with the natural gas and are mechanically separated at the wellhead. Produced water is a mixture of water vapor in the natural gas that condenses at the surface, formation water, and remnant water introduced during the hydrofracturing process. Natural gas and produced water samples are collected from the producing wells near Rulison for analysis (Figure 1).

The Colorado Oil and Gas Conservation Commission (COGCC) requires that operators with gas wells within approximately 2 miles of the Rulison site adhere to COGCC’s *Rulison Sampling and Analysis Plan for Operational and Environmental Radiological Monitoring Near Project Rulison, Revision 4* (COGCC 2017). LM, in a separate effort, has implemented the *Rulison Monitoring Plan, Revision 1* (DOE 2019a), also called the Monitoring Plan, which outlines a

strategy for sampling gas wells within 1 mile of the detonation zone. The Monitoring Plan and laboratory results from past monitoring activities are available on the LM public website at <https://www.lm.doe.gov/Rulison/Documents.aspx>. Laboratory results obtained from LM’s September 2019 monitoring event are summarized in the following sections.

Monitoring Protocol

The Monitoring Plan provides guidance on the type of samples collected (natural gas or produced water), the laboratory analyses performed, and the frequency of sample collection that is based on the amount of gas produced and the distance and direction of the well from the site. It also establishes screening levels or concentrations that, if exceeded in the sample results, require that samples be reanalyzed or additional sampling be done (DOE 2019a). The samples are analyzed for tritium, which is the most mobile contaminant remaining in significant quantities in the detonation zone. Produced water samples are submitted to a commercial environmental laboratory that provides analytical services in accordance with the *Department of Defense (DoD) Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories* (DoD/DOE 2017) to ensure that data are of known, documented quality. All laboratory results are validated according to Section 5.0, “Validation of Environmental Data,” in the *Environmental Data Validation Procedure* (LMS/PRO/S15870). Table 1 provides the sample screening levels (concentrations).

Table 1. Rulison Area Sample Screening Levels

| Analyte | Sample Matrix | Laboratory Detection Limit | Screening Concentration | Action Concentration |
|---------|----------------|----------------------------|-------------------------|---------------------------|
| Tritium | Natural gas | 10 TU ^a | 100 TU ^a | 200 TU ^a |
| | | 32 pCi/L | 320 pCi/L | 640 pCi/L |
| | Produced water | 400 pCi/L | 1000 pCi/L | 15,000 pCi/L ^b |

Notes:

The laboratory detection limits are an estimate of the laboratory’s capability of a given analytical procedure; they are reported by the laboratory as a minimum detectable concentration (MDC) that is often lower than the detection limit. The screening activities (concentrations) were obtained from the *Rulison Monitoring Plan, Revision 1* (DOE 2019a).

^a A tritium unit (TU) is equal to 3.19 pCi/L in water at a standard temperature (0 °C) and pressure (1 atmosphere).

^b The U.S. Environmental Protection Agency standard for tritium in drinking water is 20,000 pCi/L.

Abbreviations:

pCi/L = picocuries per liter

TU = tritium unit (1 tritium atom in 1×10^{18} hydrogen atoms)

Sample Collection

Samples of the produced water were collected from eight natural gas wells operating near the site on September 24, 2019 (Figure 1 and Table 2). The wells were selected based on the amount of natural gas produced and its proximity to the site (Figure 1) in accordance with the Monitoring Plan (DOE 2019a). Samples are generally collected from a well after 50 to 100 MMCF of natural gas has been produced. The last sampling events were conducted in April and July 2018 (DOE 2019b), and since those sampling events the sampled wells produced between 14 to 57 MMCF of natural gas (Table 2). No wells are currently within 0.5 mile of the site or within the designated area for natural gas samples to be collected, so only produced water samples were collected. The

produced water samples were obtained from a tap on the dump line connecting the gas-liquid separators and accumulation tank. Before sample collection, the gas-liquid separators that share a dump line were isolated using valves and then purged of produced water and condensate. The samples were contained in 1-gallon plastic containers provided by the laboratory. The produced water samples were submitted to ALS Laboratory Group in Fort Collins, Colorado, for tritium analysis. Appendix A includes a chart for each well showing the monthly and cumulative gas production volumes in MMCF with the sampling events.

Table 2. Rulison Area Natural Gas Well Sample Locations for September 2019

| Well ID | Well Pad | API No. | Natural Gas Produced Since Last Sampling Event (MMCF) | Sample Type | |
|-----------|----------|--------------|---|-------------|----------------|
| | | | | Natural Gas | Produced Water |
| BM 26-33B | 26N | 05-045-15743 | 57 | NS | Sampled |
| BM 26-33C | 26N | 05-045-15742 | 47 | NS | Sampled |
| BM 26-33D | 26N | 05-045-15739 | 56 | NS | Sampled |
| BM 26-34A | 26N | 05-045-15744 | 44 | NS | Sampled |
| BM 26-34B | 26N | 05-045-15745 | 57 | NS | Sampled |
| BM 26-34C | 26N | 05-045-15741 | 53 | NS | Sampled |
| BM 26-34D | 26N | 05-045-15748 | 43 | NS | Sampled |
| BM 36-13 | 36B | 05-045-10840 | 14 | NS | Sampled |

Abbreviation:

API = American Petroleum Institute

NS = not sampled

Sample Results

The produced water samples had no detections of tritium above the laboratory minimum detectable concentrations (MDCs) (Table 3). The laboratory results were validated in accordance with Section 5.0, “Validation of Laboratory Data,” in the *Environmental Data Validation Procedure*. All analyses were completed, and the samples were prepared and analyzed in accordance with accepted procedures for the specified methods. The laboratory radiochemical MDC reported with these data is an a priori estimate of the detection capability of a given analytical procedure; it is not an absolute concentration that can or cannot be detected. Laboratory results for produced water samples collected in September 2019 are provided in Table 3. A copy of the Data Validation Memo is provided as Appendix B.

Table 3. Produced Water Sample Results for September 2019

| Well ID | Well Pad | API No. | Tritium in Natural Gas (TU) ^a | Tritium in Produced Water (pCi/L) |
|--------------------------|----------|--------------|--|-----------------------------------|
| BM 26-33B | 26N | 05-045-15743 | NS | <400 |
| BM 26-33C | 26N | 05-045-15742 | NS | <400 |
| BM 26-33D | 26N | 05-045-15739 | NS | <400 |
| BM 26-34A | 26N | 05-045-15744 | NS | <400 |
| BM 26-34B | 26N | 05-045-15745 | NS | <400 |
| BM 26-34C | 26N | 05-045-15741 | NS | <400 |
| BM 26-34D | 26N | 05-045-15748 | NS | <400 |
| BM 36-13 | 36B | 05-045-10840 | NS | <400 |
| Screening Concentrations | | | 100 | 1000 |

Abbreviations:

API = American Petroleum Institute

NS = not sampled

pCi/L = picocuries per liter

TU = tritium unit (1 tritium atom in 1×10^{18} hydrogen atoms)

Conclusion

Natural gas wells near the Rulison site have not been impacted by detonation-related contaminants. Tritium was not detected above the laboratory MDC in any of the produced water samples collected during this sampling event (September 2019) or above screening levels in any of the produced water and natural gas samples collected during previous sampling events dating back to the first sampling event in 2007. This report is available on the LM public website at <https://www.lm.doe.gov/rulison/Sites.aspx>. Data collected during this and previous sampling events are available on the Geospatial Environmental Mapping System (GEMS) website at <https://gems.lm.doe.gov/#site=RUL>.

References

COGCC (Colorado Oil and Gas Conservation Commission), 2017. *Rulison Sampling and Analysis Plan for Operational and Environmental Radiological Monitoring Near Project Rulison, Revision 4*, July.

DoD/DOE (Department of Defense/Department of Energy), 2017. *Department of Defense (DoD) Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories*, Version 5.1, January.

DOE (U.S. Department of Energy), 2015. *United States Nuclear Tests, July 1945 through September 1992*, DOE/NV-209-Rev 16, National Nuclear Security Administration, September.

DOE (U.S. Department of Energy), 2019a. *Rulison Monitoring Plan, Revision 1*, LMS/RUL/S06178, Office of Legacy Management, December.

DOE (U.S. Department of Energy), 2019b. *Monitoring Results of Natural Gas Wells near the Rulison, Colorado, Site, April and July 2018 Monitoring Events*, LMS/RUL/S24763, Office of Legacy Management, August.

Environmental Data Validation Procedure, LMS/PRO/S15870, continually updated, prepared by Navarro Research and Engineering, Inc., for the U.S. Department of Energy Office of Legacy Management.

Reynolds, Miles, 1971. *Project Rulison—Summary of Results and Analyses*, American Nuclear Society Winter Meeting, October.

Smith, C.F., 1971. *Gas Analysis Results for Project Rulison Production Testing Samples*, Lawrence Livermore Laboratories, UCRL-51153, November.

Appendix A

Gas Well Production Data

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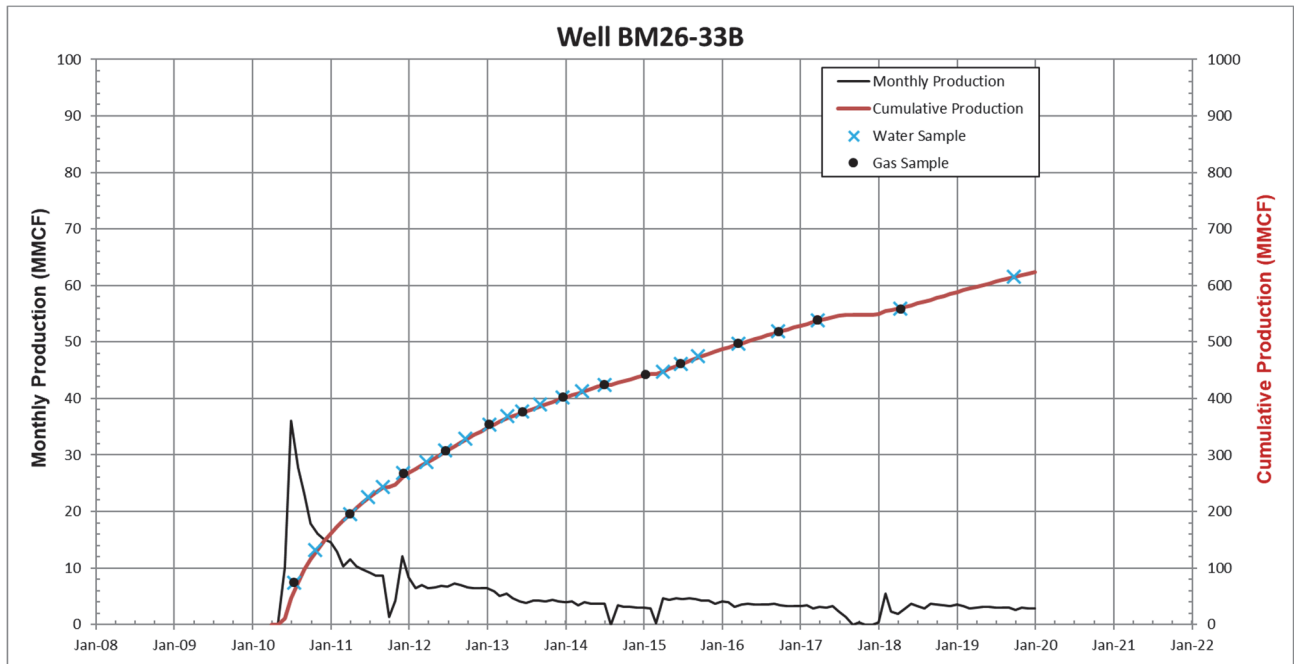


Figure A-1. Frequency of Sampling with Gas Production Data for Well BM 26-33B

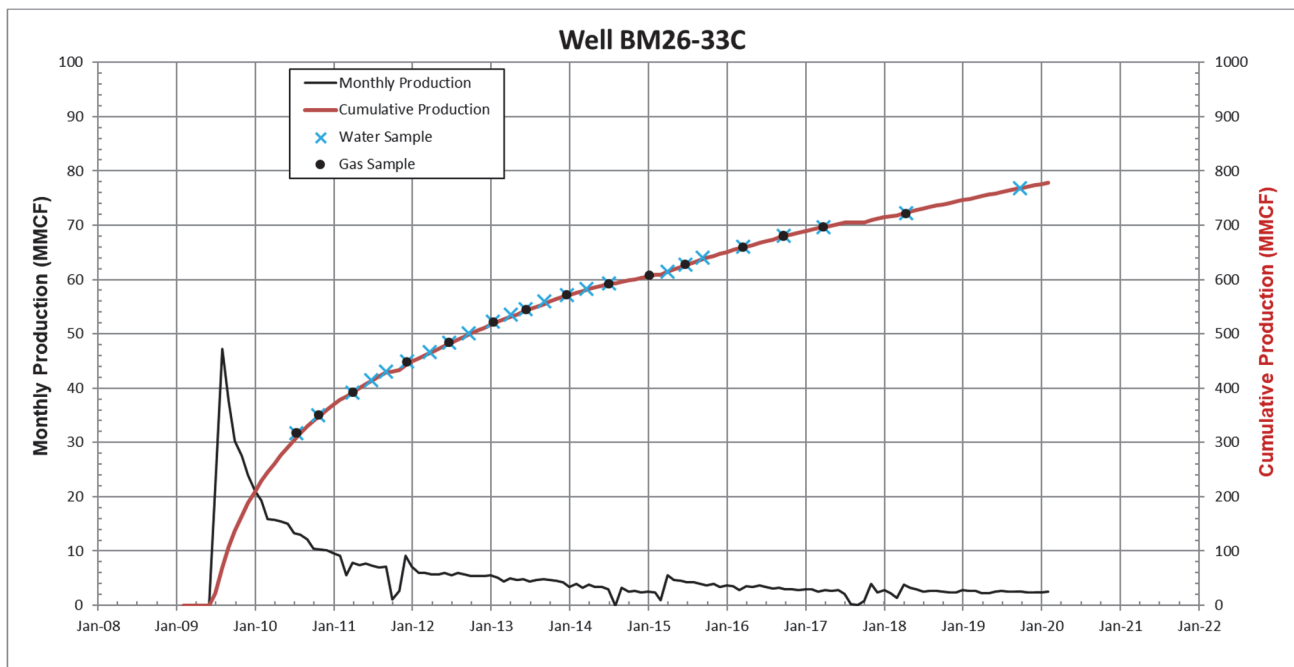


Figure A-2. Frequency of Sampling with Gas Production Data for Well BM 26-33C

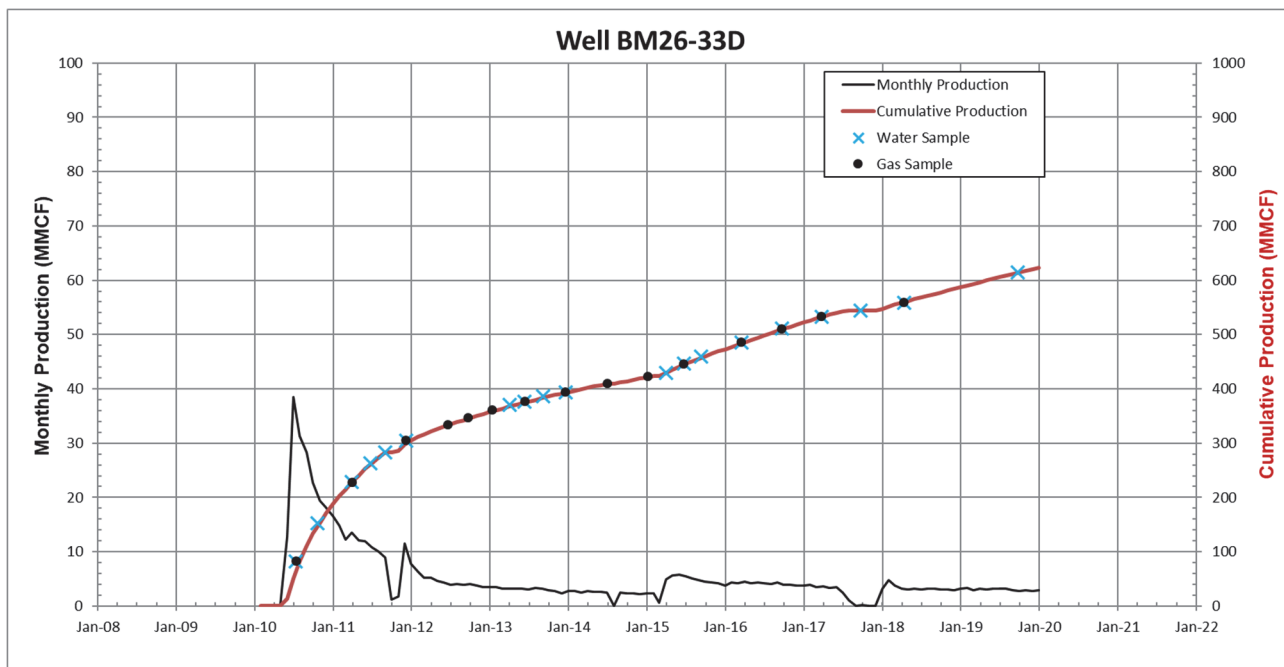


Figure A-3. Frequency of Sampling with Gas Production Data for Well BM 26-33D

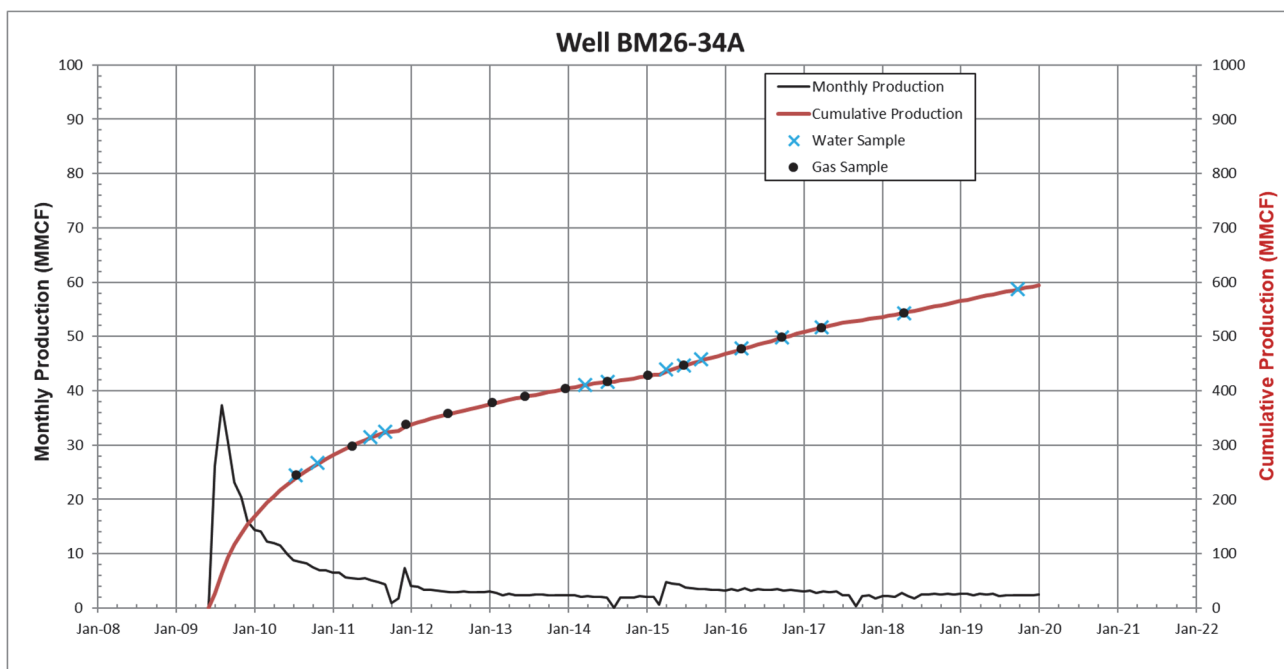


Figure A-4. Frequency of Sampling with Gas Production Data for Well BM 26-34A

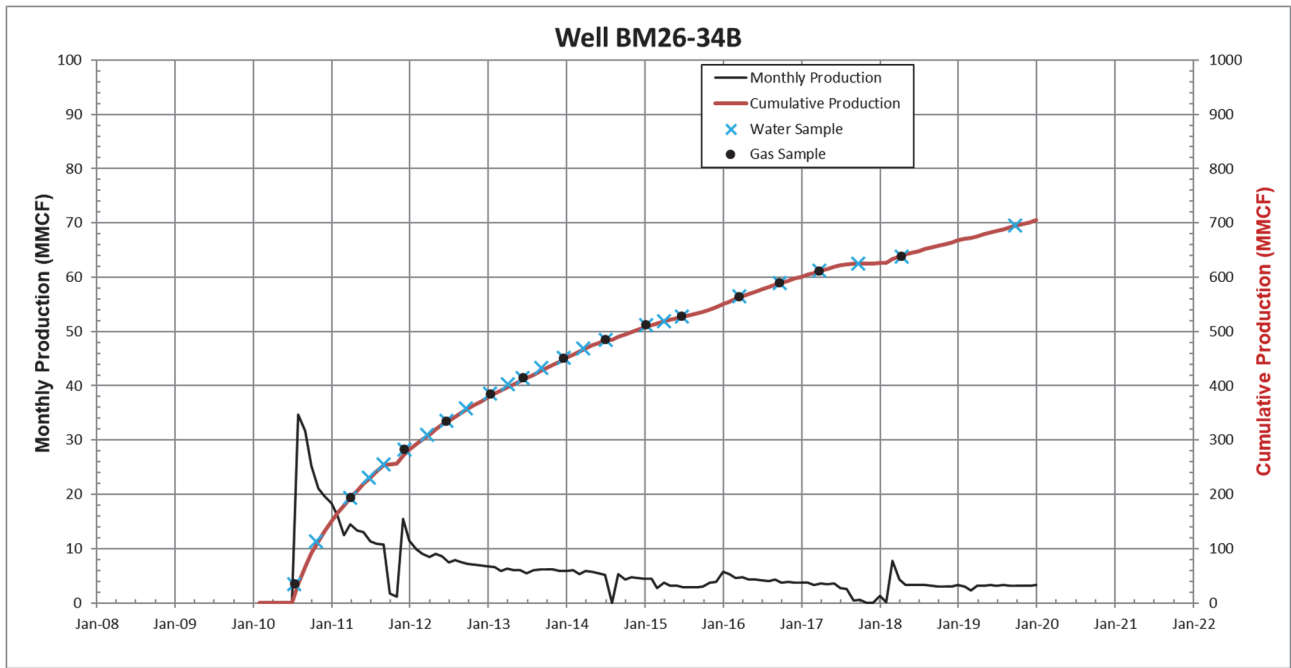


Figure A-5. Frequency of Sampling with Gas Production Data for Well BM 26-34B

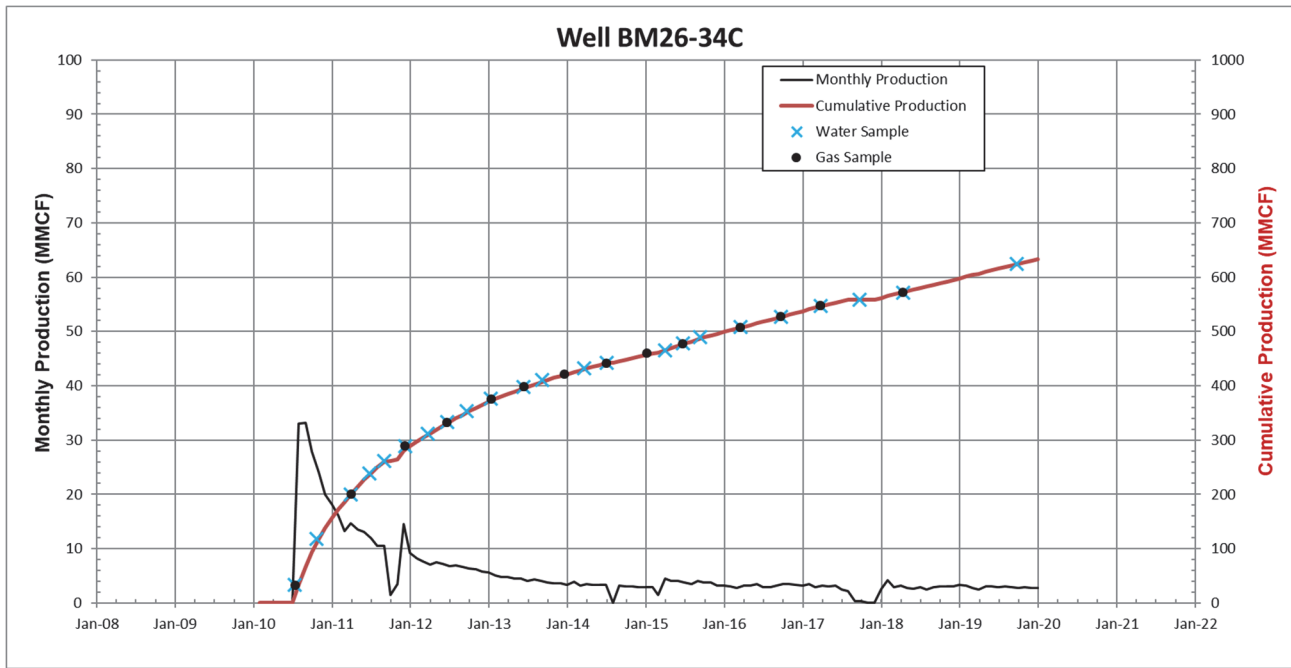


Figure A-6. Frequency of Sampling with Gas Production Data for Well BM 26-34C

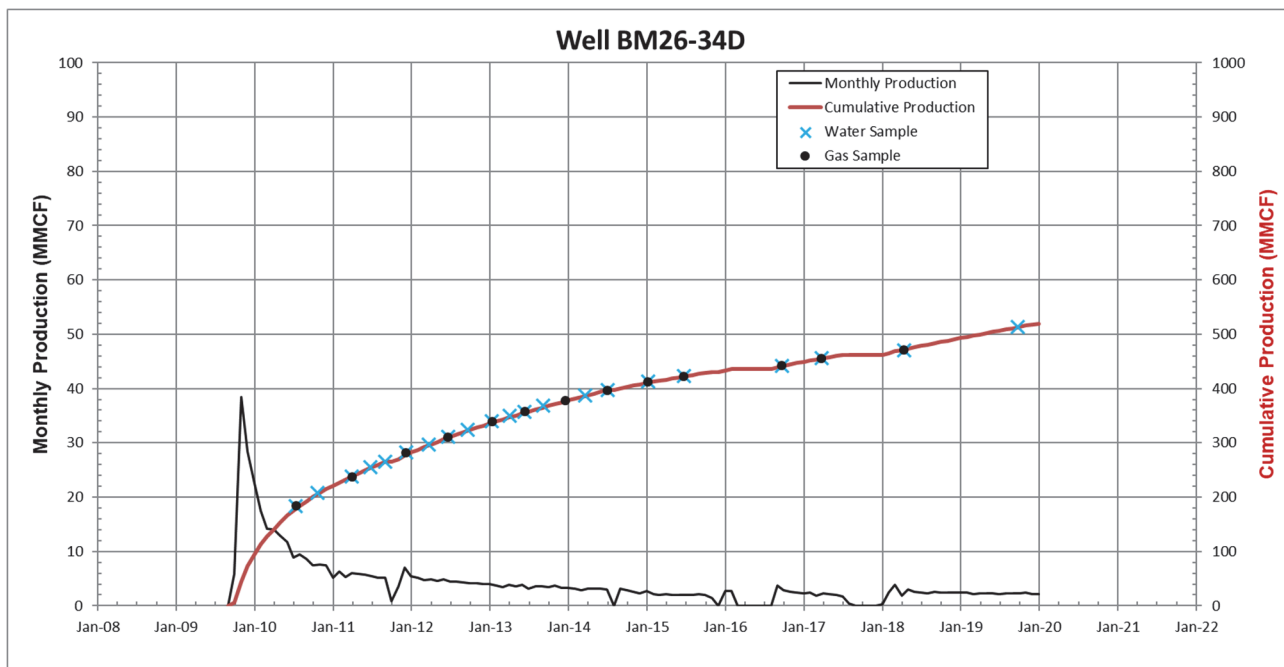


Figure A-7. Frequency of Sampling with Gas Production Data for Well BM 26-34D

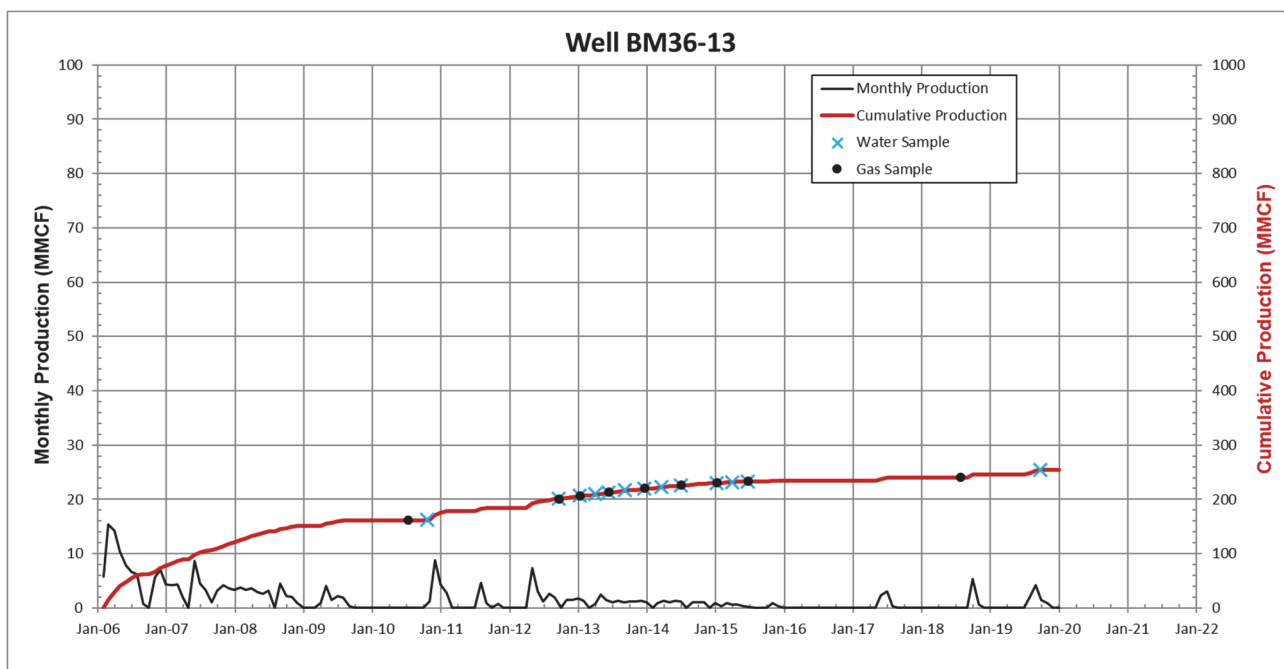


Figure A-8. Frequency of Sampling with Gas Production Data for Well BM 36-13

Appendix B

Data Validation Memo

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To: Rick Findlay, Navarro
From: Stephen Donovan, Navarro
CC: Janice McDonald, Navarro
Date: April 16, 2020
Re: Validation of September 2019 Produced Water Data from the Rulison Site

Validation of data generated from the September 2019 produced water sampling event at the Rulison, Colorado, Site has been completed. This Level 2 validation was conducted according to the *Environmental Data Validation Procedure* (LMS/PRO/S15870).

The samples were submitted for analysis identified by Task Code RUL01-02.1909004. Planned monitoring locations are shown in the Sampling and Analysis Work Order (Enclosure 1). Produced water samples were collected from 8 of the 15 planned sample locations. The sample locations were reduced to align with the Rulison Monitoring Plan, Revision 1 and discussions with the Colorado Oil and Gas Conservation Commission. See the Trip Report (Enclosure 2) for additional details.

All environmental data from this sampling event are considered validated and available for use. Site data is available for viewing with dynamic mapping via the GEMS (Geospatial Environmental Mapping System) website at <http://gems.lm.doe.gov/#>. The Field Data Assessment (Enclosure 3) includes discussion of the field data and field quality control samples. The Laboratory Performance Assessment (Enclosure 4) documents the review of the laboratory data. Summaries of Enclosures 3 and 4 are presented below.

Sampling and Analysis Work (Enclosure 1)

Trip Report (Enclosure 2)

Field Data Assessment (Enclosure 3)

Verification of Field Activities

A Field Activities Verification Checklist was completed with no issues identified.

Assessment of Field Quality Control Samples

A duplicate sample was collected from location 05-045-15748. The duplicate results met the acceptance criteria, demonstrating acceptable precision.

Laboratory Performance Assessments (Enclosure 4)

All laboratory analytical quality control criteria were met. Analytical data and the associated qualifiers can be viewed in reports from the environmental database.

Enclosures (4)

Enclosure 1
Sampling and Analysis Work Order



September 9, 2019

Task Assignment 104
Control Number 19-1661

U.S. Department of Energy
Office of Legacy Management
ATTN: Ms. Jalena Dayvault
LM Site Manager
2597 Legacy Way
Grand Junction, CO 81503

SUBJECT: Contract No. DE-LM0000421, Navarro Research and Engineering, Inc.
(Navarro)
Task Assignment 104, LTS&M - Nevada Off Sites and Monticello Site
September 2019 Environmental Sampling at the Rulison, Colorado, Site

REFERENCE: Task Assignment 104, 1-104-1-04-619, Rulison, Colorado, Site

Dear Ms. Dayvault:

The purpose of this letter is to inform you of the upcoming sampling event at the Rulison, Colorado, Site. Enclosed are the map and tables specifying sample locations and analytes for produced water and gas monitoring at the site. Data will be collected at this site as part of the routine gas well sampling currently scheduled to begin the week of September 23, 2019.

The following list shows the locations scheduled for sampling during this event.

PRODUCED WATER MONITORING WELLS

| | |
|--------------------------|--------------------------|
| BM 26-22B (05-045-16086) | BM 26-34C (05-045-15741) |
| BM 26-22C (05-045-16087) | BM 26-34D (05-045-15748) |
| BM 26-22D (05-045-16074) | BM 35-32A (05-045-10919) |
| BM 26-33B (05-045-15743) | BM 36-13 (05-045-10840) |
| BM 26-33C (05-045-15742) | BM 36-13A (if available) |
| BM 26-33D (05-045-15739) | BM 36-13B (05-045-15469) |
| BM 26-34A (05-045-15744) | BM 36-13C (if available) |
| BM 26-34B (05-045-15745) | |

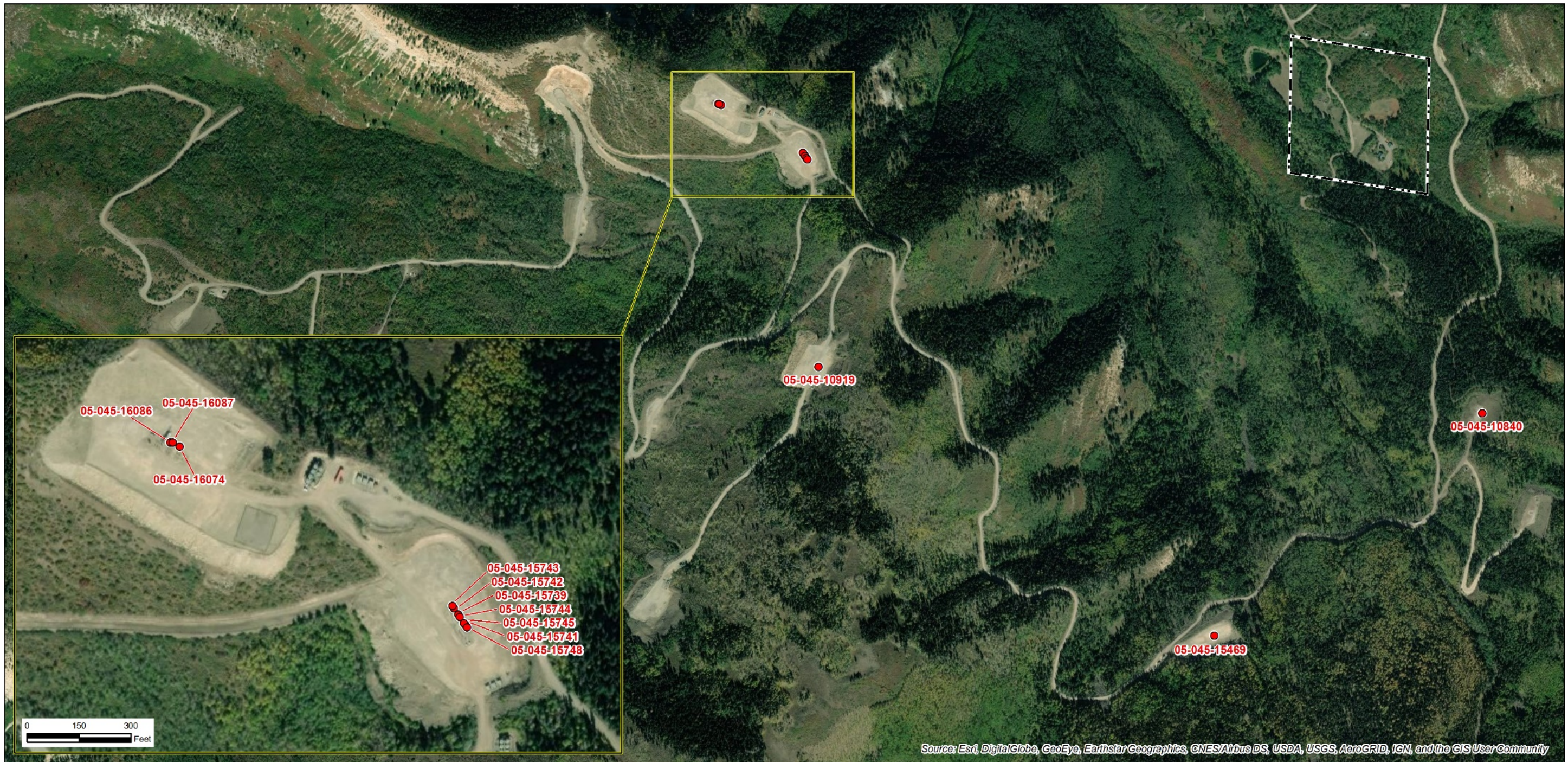
All samples will be collected as directed in the *Sampling and Analysis Plan for U.S. Department of Energy Office of Legacy Management Sites*. Notification for access to locations on private property will be conducted prior to the beginning of fieldwork.

**Sampling Frequencies for Locations at
Rulison, Colorado**

| Location ID | Quarterly | Semiannually | Annually | Biennially | Not Sampled | Notes |
|-------------------------------------|-----------|--------------|----------|------------|-------------|--------------|
| Gas and Produced Water Wells | | | | | | |
| BM 26-22B | | X | | | | 05-045-16086 |
| BM 26-22C | | X | | | | 05-045-16087 |
| BM 26-22D | | X | | | | 05-045-16074 |
| BM 26-33B | | X | | | | 05-045-15743 |
| BM 26-33C | | X | | | | 05-045-15742 |
| BM 26-33D | | X | | | | 05-045-15739 |
| BM 26-34A | | X | | | | 05-045-15744 |
| BM 26-34B | | X | | | | 05-045-15745 |
| BM 26-34C | | X | | | | 05-045-15741 |
| BM 26-34D | | X | | | | 05-045-15748 |
| BM 35-32A | | X | | | | 05-045-10919 |
| BM 36-13 | | X | | | | 05-045-10840 |
| BM 36-13A | | X | | | | If available |
| BM 36-13B | | X | | | | 05-045-15469 |
| BM 36-13C | | X | | | | If available |

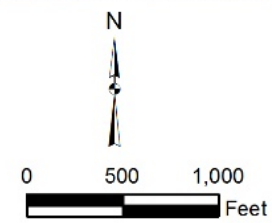
Sampling based on amount of gas produced

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LEGEND

- Natural Gas Well to be Sampled
- ⋮ Site Boundary



U.S. DEPARTMENT OF ENERGY
OFFICE OF LEGACY MANAGEMENT

Work Performed by
Navarro Research & Engineering, Inc.
Under DOE Contract Number DE-LM0000421

Produced Water and Gas
Planned Sample Locations
Rulison, CO, Site
September 2019

| | |
|-----------------------------------|------------------------------|
| DATE PREPARED: August 26, 2019 | FILE NAME: S2647602-11x17 |
|-----------------------------------|------------------------------|

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Enclosure 2
Trip Report

memo



To: Rick Findlay, Navarro
From: Jeff Price, Navarro
Date: October 14, 2019
CC: Steve Donovan, Navarro
Rex Hodges, Navarro
EDD Delivery
Re: Trip Report – 2019 Gas Well Sampling Event

Site: Rulison, Colorado, Site

Date of Event: September 24, 2019

Team Members: Jeff Price and Rick Findlay (Navarro).

Number of Locations Sampled: Produced water samples were collected for tritium analysis from eight natural gas wells as prescribed in the *Rulison Monitoring Plan, Revision 1* (LMS/RUL/S06178).

Locations Not Sampled/Reason: All the natural gas wells were sampled as prescribed in the *Rulison Monitoring Plan, Revision 1* (LMS/RUL/S06178).

Quality Control Sample Cross Reference: The following is the false identification assigned to the quality control sample.

| False Location | False Sample ID | Parent Location | Parent Sample ID | Sample Type | Associated Matrix |
|----------------|----------------------|-----------------|----------------------|-------------|-------------------|
| 2487 | RUL01-02.1909004-016 | 05-045-15748 | RUL01-02.1909004-010 | Duplicate | Produced Water |

Task Code Assigned: Samples were assigned to Task Codes RUL01-02.1807003. Field data sheets can be found at <\\crow\SMS\RUL01-02.1909004\RECORDS\FieldData>. Samples were shipped via FedEx from Grand Junction on September 26, 2019, to ALS Laboratory Group in Fort Collins, CO.

Well Inspection Summary: No issues were identified.

Sampling Method: Samples were collected according to the *Rulison Monitoring Plan, Revision 1* (LMS/RUL/S06178) and the *Sampling and Analysis Plan (SAP) for the U. S. Department of Energy Office of Legacy Management Sites* (LMS/PRO/S04351, continually updated) and Program Directive RUL-2015-01.

Field Variance: None.

Rick Findlay
October 14, 2019
Page 2

Equipment: All equipment functioned properly.

Stakeholder/Regulatory/DOE: K. Rice with Caerus Oil and Gas provided access to the natural gas wells.

Safety Issues: None.

Access Issues: None.

General Information: Nothing to note.

Immediate Actions Taken: None.

Future Actions Required or Suggested: None.

Enclosure 3
Field Data Assessment

Water Sampling Field Activities Verification Checklist

| Project | <u>Rulison, Colorado, Site</u> | Date(s) of Water Sampling | <u>September 24, 2019</u> |
|--|----------------------------------|--|---------------------------|
| Date(s) of Verification | <u>April 14, 2020</u> | Name of Verifier | <u>Stephen Donovan</u> |
| | | Response (Yes, No, NA) | Comments |
| 1. Is the SAP the primary document directing field procedures? List any Program Directives or other documents, SOPs, instructions. | Yes | | |
| 2. Were the sampling locations specified in the planning documents sampled? | No | Sample locations were reduced to align with the Rulison Monitoring Plan, Revision 1. | |
| 3. Were field equipment calibrations conducted as specified in the above-named documents? | NA | Field measurements were not required. | |
| 4. Was an operational check of the field equipment conducted daily? Did the operational checks meet criteria? | NA NA | | |
| 5. Were the number and types (alkalinity, temperature, specific conductance, pH, turbidity, DO, ORP) of field measurements taken as specified? | NA | | |
| 6. Were wells categorized correctly? | NA | This sampling event did not include groundwater. | |
| 7. Were the following conditions met when purging a Category I well: Was one pump/tubing volume purged prior to sampling? Did the water level stabilize prior to sampling? Did pH, specific conductance, and turbidity measurements meet criteria prior to sampling? Was the flow rate less than 500 mL/min? | NA NA NA NA | This sampling event did not include groundwater. This sampling event did not include groundwater. | |

Water Sampling Field Activities Verification Checklist (continued)

| | Response (Yes, No, NA) | Comments |
|--|---------------------------|--|
| 8. Were the following conditions met when purging a Category II well: | | |
| Was the flow rate less than 500 mL/min? | NA | This sampling event did not include groundwater. |
| Was one pump/tubing volume removed prior to sampling? | NA | |
| 9. Were duplicates taken at a frequency of one per 20 samples? | Yes | A duplicate sample was collected at location 05-045-15748. |
| 10. Were equipment blanks taken at a frequency of one per 20 samples that were collected with non-dedicated equipment? | NA | An equipment blank was not required. |
| 11. Were trip blanks prepared and included with each shipment of VOC samples? | NA | |
| 12. Were the true identities of the QC samples documented? | Yes | |
| 13. Were samples collected in the containers specified? | Yes | |
| 14. Were samples filtered and preserved as specified? | Yes | |
| 15. Were the number and types of samples collected as specified? | Yes | |
| 16. Were chain of custody records completed and was sample custody maintained? | Yes | |
| 17. Was all pertinent information documented on the field data sheets? | Yes | |
| 18. Was the presence or absence of ice in the cooler documented at every sample location? | NA | Sample cooling was not required. |
| 19. Were water levels measured at the locations specified in the planning documents? | NA | |

Data Qualifier Summary

None of the analytical results were qualified based on field quality control.

Field Duplicate Assessment

Field duplicate samples are collected and analyzed as an indication of overall precision of the measurement process. The precision observed includes both field and laboratory precision and has more variability than laboratory duplicates, which measure only laboratory performance. A duplicate sample was collected from location 05-045-15748. For radiochemical measurements, the relative error ratio (the ratio of the absolute difference between the sample and duplicate results and the sum of the 1-sigma uncertainties) is used to evaluate duplicate results and should be less than 3. All duplicate results met these criteria demonstrating acceptable precision.

Validation Report: Field Duplicates

Page 1 of 1
20-Nov-2019

Project: Rulison Produced Water **Task Code:** RUL01-02.1909004 **Lab Code:** PAR

| Analyte | Duplicate: RUL01-02.1909004-016 | | | | Sample: RUL01-02.1909004-010 05-045-15748 | | | | RPD | RER | Units |
|---------|---------------------------------|------------|---------|----------|--|------------|---------|----------|-----|-----|-------|
| | Result | Qualifiers | Uncert. | Dilution | Result | Qualifiers | Uncert. | Dilution | | | |
| Tritium | 57.2 | U | 115 | 1 | -117 | U | 88.8 | 1 | | 2.3 | pCi/L |

QC Checks: RPD: Relative Percent Difference RER: Relative Error Ratio

Enclosure 4
Laboratory Performance Assessment

General Information

Task ID: RUL01-02.1909004
Sample Event: September 24, 2019
Site(s): Rulison, Colorado, Site
Laboratory: ALS Laboratory Group, Fort Collins, Colorado
Work Order No.: 1909573
Analysis: Radiochemistry
Validator: Stephen Donivan
Review Date: November 21, 2019

This validation was performed according to the *Environmental Data Validation Procedure* (LMS/PRO/S15870). The procedure was applied at Level 2, Data Verification. See attached Data Validation Worksheets for supporting documentation on the data review and validation. All analyses were successfully completed. The samples were prepared and analyzed using accepted procedures based on methods specified by line item code, which are listed in Table 1.

Table 1. Analytes and Methods

| Analyte | Line Item Code | Prep Method | Analytical Method |
|---------|----------------|--------------|-------------------|
| Tritium | LCS-A-001 | PA SOP700R10 | PA SOP704R9 |

Data Qualifier Summary

None of the analytical results required qualification.

Sample Shipping/Receiving

ALS Laboratory Group in Fort Collins, Colorado, received nine water samples on September 27, 2019, accompanied by a Chain of Custody form. The Chain of Custody form was checked to confirm that the samples were listed with sample collection dates and times, and that signatures and dates were present indicating sample relinquishment and receipt. Copies of the shipping labels were included in the receiving documentation. The Chain of Custody form was complete with no errors or omissions.

Preservation and Holding Times

The sample shipment was received intact at ambient temperature which complies with requirements. All samples were received in the correct container types and had been preserved correctly for the requested analyses. Sample analysis was completed within the applicable holding times.

Detection and Quantitation Limits

The method detection limit (MDL) was reported for all metal and wet chemical analytes as required. The MDL, as defined in Title 40 *Code of Federal Regulations* Section 136, is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The practical quantitation limit (PQL) for these analytes is the lowest concentration that can be reliably measured and is defined as 5 times the MDL.

For radiochemical analytes (those measured by radiometric counting) the MDL and PQL are not applicable, and these results are evaluated using the minimum detectable concentration (MDC), the decision level concentration (DLC), and the determination limit (DL). The DLC is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is estimated as 3 times the 1-sigma total propagated uncertainty. Results that are greater than the MDC but less than the DLC are qualified with a U flag as not detected. The DL for radiochemical results is the lowest concentration that can be reliably measured and is defined as 3 times the MDC. Results that were not previously U qualified and are less than the DL are qualified with a J flag as estimated values.

The reported MDCs for radiochemical analytes met the detection limits requirements with the following exception. The required detection limits were not met for the gross alpha result because of the elevated levels of dissolved solids in the samples.

Laboratory Instrument Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for all analytes. Initial calibration verification demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run. Compliance requirements for continuing calibration checks are established to ensure that the instrument continues to be capable of producing acceptable qualitative and quantitative data. All laboratory instrument calibrations were performed correctly in accordance with the cited methods. All calibration and laboratory spike standards were prepared from independent sources.

Method and Calibration Blanks

Method blanks are analyzed to assess any contamination that may have occurred during sample preparation. Calibration blanks are analyzed to assess instrument contamination prior to and during sample analysis. All radiochemical method blank results were below the decision level concentration.

Laboratory Control Sample

Laboratory control samples were analyzed at the correct frequency to provide information on the accuracy of the analytical method and the overall laboratory performance, including sample preparation. All control sample results were acceptable.

Laboratory Replicate Analysis

Laboratory replicate sample results demonstrate acceptable laboratory precision. The relative percent difference value for the chloride matrix spike replicate met the acceptance criteria. The radiochemical relative error ratio (calculated using the one-sigma total propagated uncertainty) for the sample replicates was less than three for all replicates.

Matrix Spike Analysis

Matrix spike and matrix spike duplicate (MS/MSD) samples are used to measure method performance in the sample matrix. The MS/MSD data are not evaluated when the concentration

of the unspiked sample is greater than four times the spike concentration. The spike recoveries were acceptable for all analytes.

Completeness

Results were reported in the correct units for all analytes requested using contract-required laboratory qualifiers.

Electronic Data Deliverable (EDD) File

The EDD file arrived on October 25, 2019. The EDD was examined to verify that the file was complete and in compliance with requirements. The contents of the file were compared to the requested analyses to ensure all and only the requested data were delivered. The contents of the EDD were manually examined to verify that the sample results accurately reflect the data contained in the sample data package.

General Data Validation Report

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Task Code: RUL01-02.1909004 **Lab Code:** PAR **Validator:** Stephen Donovan **Validation Date:** 11-20-2019

Project: Rulison Produced Water

Samples: 9

Analysis Type: General Chemistry Metals Organics Radiochemistry

Chain of Custody

Sample

| | |
|---|---|
| Present: <u>OK</u> Signed: <u>OK</u> Dated: <u>OK</u> | Integrity: <u>OK</u> Preservation: <u>OK</u> Temperature: <u>OK</u> |
|---|---|

Check

Summary

| | |
|--------------------------|---|
| Holding Times: | All analyses were completed within the applicable holding times. |
| Detection Limits: | The reported detection limits are equal to or below the contract required limits. |
| Field Duplicates: | There was 1 duplicate evaluated. |

Radiochemistry Data Validation Worksheet

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20-Nov-2019

Task Code: RUL01-02.1909004

Project: Rulison Produced Water

Lab Code: PAR

| Sample ID | Analyte | Analysis Date | QC Type | Result Type | Result | Flag | TPU | Spike Recovery | Spike Dup Recovery | Lower Limit | Upper Limit | RPD | RPD Limit | RER | Comments |
|----------------------|---------|---------------|---------|-------------|----------|------|------|----------------|--------------------|-------------|-------------|-----|-----------|-------|----------|
| | Tritium | 10-16-2019 | LCS | SC | 16100.00 | | 2560 | 97.90 | | 85 | 115 | | | | |
| | Tritium | 10-16-2019 | MB | TRG | -116.00 | U | 88.8 | | | | | | | | |
| RUL01-02.1909004-004 | Tritium | 10-16-2019 | MS | SC | 15400.00 | | 2440 | 93.00 | | 85 | 115 | | | | |
| RUL01-02.1909004-004 | Tritium | 10-16-2019 | R | TRG | -79.20 | U | 94.6 | | | | | | | 0.815 | |

QC Types: LCS: Laboratory Control Sample LCSD: Laboratory Control Sample Duplicate MB: Method Blank MS: Matrix Spike MSD: Matrix Spike Duplicate R: Replicate

Result Types: IS: Internal Standard SC: Spike Analyte TRG: Target analyte

QC Checks: RPD: Relative Percent Difference RER: Relative Error Ratio TPU: Total Propagated Uncertainty