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REVISION/CHANGE LOG			
Revision/Change Letter	Description of Changes	Pages Affected	Date of Revision/Change
FR0	Initial Bluesheeting	All	10/20/2017
FR1	Non-Intent Revision to Incorporate Bluesheeting Changes	All	1/3/2018
FR2	Revised to include an additional form and changes to section 6.8.34 and 9.1 to reflect the form change.	16 & 17	6/27/2018
FR3	General Revision	All	8/29/2019
FR4	General formatting. Updated Section 4.0, 5.0 and 7.0. Removed Section 6.1. Addition of Steps 6.3.44-6.4.49.	All	6/4/2020
FR4A	Updated Step 6.3.22B to say Proceed to Step 6.3.64.	10	10/6/2020
FR4B	Periodic Review had been completed with no changes identified in procedure technical content. Nonintent change to correct SME, sprover and dates has been incorporated per CP3-NS-2001. Date for review cycle has been reset.	All	9/13/2021
FR4C	Periodic Review has been completed with no changes identified in procedure technical content. Nonintent changes have been incorporated per CP3-NS-2001. Date for review cycle has been reset.	All	11/04/2024

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1.0 PURPOSE AND SCOPE

1.1 Purpose

To define steps necessary for the calibration and operation of the Trichloroethene (TCE) On-Line Analyzer for the Northwest Plume Groundwater System.

1.2 Scope

This procedure applies to Paducah Gaseous Diffusion Plant (PGDP) Deactivation & Remediation (D&R) Contractor Environmental Remediation Personnel performing activities associated with the Continuous Monitoring System (CMS) -5000 TCE On-Line Analyzer.

Sections of this procedure may be performed independently or concurrently as directed by an Activity Level Work Control Document (ALWCD).

2.0 REFERENCES

2.1 Use References

- CP2-ER-0046, *Paducah Plume Operations Maintenance, Sampling and Analysis, and Calibration and Testing Plan*
- CP3-OP-0207, *Use of Procedures*
- CP3-SM-0049, *Installed Plant Instrumentation Measuring and Test Equipment*
- CP4-ER-0017, *Northwest/Northeast (NW/NE) Plume Daily Operational Data Collection and Maintenance*

2.2 Source References

- CP3-SM-1101, *Work Package Development*
- Department of Energy (DOE)/OR/07-1253, *Operations and Maintenance Plan for the Northwest Plume Groundwater Containment System Interim Remedial Action Plan at PGDP Paducah, Kentucky*
- Inficon CMS5000™, *Monitoring System Operating Manual*
- Job Hazard Analysis (JHA)-10844 *Northwest/Northeast (NW/NE) Plume Daily Operational Data Collection and Maintenance*

3.0 COMMITMENTS

- In accordance with CP2-ER-0046, *Paducah Plume Operations Maintenance, Sampling and Analysis, and Calibration and Testing Plan*, the TCE on-line analyzer at the C-612 facility requires semi-annual analytical method calibration unless the check standard multiplier is Out of Tolerance (OOT).
- In accordance with CP2-ER-0046, the TCE on-line analyzer at the C-612 facility requires routine quarterly TCE online analyzer check standard method calibration.
- In accordance with CP2-ER-0046, the TCE on-line analyzer at the C-612 facility requires routine semiannual alarm testing to verify analyzer performance.

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4.0 PRECAUTIONS AND LIMITATIONS

4.1 Precautions

- 4.1.1 Safety glasses with side shield shall be worn.
- 4.1.2 **When** handling **or** mixing TCE Calibration Standard, **then** Supreno EC Microflex Nitrile, Showa 730, or Silver Shield chemical gloves shall be worn.
- 4.1.3 Personnel shall be made familiar with the hazards associated with exposure to TCE and review Safety Data Sheet (SDS).
- 4.1.4 **When** performing mixing activity, **then** general building ventilation for C-612 shall be utilized.
- 4.1.5 **When NOT** in use, **then** cylinder valves shall be CLOSED.
- 4.1.6 Cylinders shall be labeled with full/in service/empty tags to indicate the status of the cylinder.
- 4.1.7 **When** cylinders are **NOT** in use or at the end of each work shift, **then** regulators shall be removed from cylinder **and** the valve protection caps shall be re-installed.
- 4.1.8 Cylinder shall be inspected before use and **if** the cylinder or associated equipment (regulator, hose, etc.), is defective, **then** the equipment shall be removed from service and tagged as defective in accordance with the Accident Prevention/Equipment Control Tag program document.
- 4.1.9 Cylinders shall be stored away from sources of heat and flammable substances.
- 4.1.10 Cylinders shall be stored in an upright position and secured by chains or other means to prevent them from being knocked over.
- 4.1.11 Cylinder carts shall be used to transport cylinders.
- 4.1.12 Cylinders shall **NOT** be rolled by hand.

4.2 Limitations

- 4.2.1 The breakthrough time of Supreno EC Microflex Nitrite gloves is 2 hours for TCE.
- 4.2.2 Supreno EC Microflex Nitrile gloves are **NOT** to be used for more than 2 hours after contact with TCE contaminated liquid.
- 4.2.3 The breakthrough time of Showa 730 gloves is 4 hours for TCE.
- 4.2.4 Showa 730 gloves are **NOT** to be used for more than 4 hours after contact with TCE contaminated liquid.

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5.0 PREREQUISITES

- 5.1 Prior to performing any action steps identified in this procedure for the first time, review this document based upon its level of use according to CP3-OP-0207, *Use of Procedures*.
- 5.2 Before executing this procedure, obtain approval of the NW/NE Pump and Treat Project Manager or designee.
- 5.3 If the On-Line analyzer is unable to complete calibration per the procedure, **then** Engineering will have the authority to direct that sections be repeated as necessary to facilitate troubleshooting.
- 5.4 If new or temporary actions will require further documentation for any OOT other than the calibration form if necessary, **then** Engineering will provide direction.

6.0 INSTRUCTIONS

6.1 Check of Argon Gas

Operations Personnel

- 6.1.1 Check the pressure of the argon gas cylinder.
- 6.1.2 If the pressure is greater than 600 pounds per square inch gauge (psig), **then** proceed to Step 6.1.18.
- 6.1.3 If cylinder pressure is less than 600 psig, **then** notify the NW/NE Pump and Treat Project Manager.

CAUTION:

Argon supply must be ultra-high purity (99.999%) for optimal operation. Failure to use the recommended argon purity will result in poor instrument performance and possible instrument damage.

- 6.1.4 If the analyzer is performing a sample run, **then** wait until it is complete and the instrument is in the “Sleeping” mode.
- 6.1.5 **When** the instrument is in the “Sleeping” mode, **then** take the instrument out of automatic operation by pressing the “Esc” key.

NOTE:

Loss of argon pressure to the analyzer will result in warning messages appearing on the analyzer display. This is normal during cylinder replacement and will clear once the new argon supply is established to the analyzer.

Operations Personnel

- 6.1.6 Close the cylinder valve.
- 6.1.7 Disconnect copper argon supply line from the left side of the analyzer using the quick connect fitting.
- 6.1.8 Remove the pressure regulator from the cylinder.

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- 6.1.9 Attach the cylinder cap to the empty cylinder.
- 6.1.10 Remove the empty cylinder.
- 6.1.11 Secure the new cylinder.
- 6.1.12 Remove the cylinder cap from the new cylinder.
- 6.1.13 Place the pressure regulator on the new cylinder.
- 6.1.14 Open the cylinder valve.
- 6.1.15 Ensure the pressure of the argon gas cylinder is greater than 600 psig.
- 6.1.16 Depress argon supply line quick connect fitting on clean surface for a minimum of 5 seconds to purge supply line.
- 6.1.17 Reconnect copper argon supply line to the analyzer.
- 6.1.18 **If** the analyzer is to be restarted, **then** proceed to Section **6.6** to run normal operational methods.
- 6.1.19 **If** analyzer is to be calibrated, **then** proceed to Section **6.2** to perform calibration methods.

6.2 TCE On-Line Analyzer Check Standard Method Calibration

Instrument Technician

- 6.2.1 **If** the analyzer is performing a sample run, **then** wait until it is complete and the instrument is in the “Sleeping” mode.
- 6.2.2 **When** the instrument is in the “Sleeping” mode, **then** take the instrument out of automatic operation by pressing the “Esc” key on the analyzer.
- 6.2.3 Connect laptop to analyzer utilizing either the Ethernet or Wi-Fi connection.
- 6.2.4 Open CMS IQ software.
- 6.2.5 Select the analyzer icon located at the bottom of the page to enter analyzer system setup.
- 6.2.6 Double click the “Status” icon.
- 6.2.7 Select the “Parameters” tab.
- 6.2.8 Document current “CkStd Multiplier” value in the as-found field on form CP4-ER-0019-F01, *NWP Groundwater System Trichloroethene On-Line Analyzer Calibration*.
- 6.2.9 **If** the “CkStd Multiplier” value is greater than or equal to (\geq) 0.9 and less than or equal to (\leq) 1.1, **then** proceed to Section **6.6** **or** return to section(s) **6.3** **or** **6.7** if performing these sections.

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- 6.2.10 If the “CkStd Multiplier” value is less than or equal to (\leq) 0.7 or greater than or equal to (\geq) 1.3, **then** notify the NW/NE Pump and Treat Project Manager prior to proceeding with check standard calibration.
- 6.2.11 Return to the analyzer system setup screen in the CMS IQ software on the laptop.
- 6.2.12 Double click “Run Method” icon.
- 6.2.13 Select the “Ck Std.mth” file and click OK.
- 6.2.14 Allow for analyzer temperatures to stabilize and the check standard method to run.

NOTE:

Three consecutive check standard methods are needed to perform this calibration.

- 6.2.15 Repeat Steps 6.2.12 through 6.2.14 until a total of three check standard methods have been ran.
- 6.2.16 After three consecutive check standards methods have been ran, close the “Run Method” window.
- 6.2.17 Click on the “Calibrate” button on the CMS IQ toolbar.
- 6.2.18 Double click the “Ck Std.mth” method to open the “Calibrate” window.

NOTE:

Data files names are generated by the sample methods with the following naming scheme:

“SMN_YYYYMMDD_XX”

Where

SMN = Sample Method Name (e.g. “Ck Std”, “Water Purge TCE”, etc.)

YYYYMMDD = Calendar Year, Month, and Day

XX = Sequential Number (starting at “01” and resets every day)

- 6.2.19 Select the first data file found in the “Data Files” subsection.
- 6.2.20 Press “Delete” on the keyboard until all data files have been removed.
- 6.2.21 Once the old data files have been removed, click on the “Browse” button in the “Data Files” subsection to open the “Select Data Files” window.
- 6.2.22 Double click the “Ck Std” folder to open.
- 6.2.23 Select the most recent “Ck Std” file as indicated by the timestamp for the data file.
- 6.2.24 Hold “Shift” on the keyboard **and** select the third “Ck Std” file to highlight the three most recent files and click “OK.”
- 6.2.25 **When** the “Methods Differ” window opens for each file, **then** click “Yes” to acknowledge the window for each of the three data files.

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- 6.2.26 **When** the “Inconsistent Concentrations” warning appears, **then** click “OK” to acknowledge the warning.
- 6.2.27 Select the “Conc/Factor” field (may appear as “Conc/Fa...”) for first data file found in the “Data Files” subsection.
- 6.2.28 Enter a value of “1” in to this field.
- 6.2.29 Repeat Step 6.2.27 and 6.2.28 for each of the remaining data files.
- 6.2.30 Check the checkbox next to each data file found in the “Select” field (may appear as “Sel...”).
- 6.2.31 Check the box next to “Reset Library” to remove old calibration data from the calibration library.
- 6.2.32 Click “Start” to recalibrate the “Ck Std” method.

NOTE:

“Relative Standard Deviation %” value can be found in the bottom right hand corner of the “Calibrate” window.

- 6.2.33 **If** the “Relative Standard Deviation %” value is greater than or equal to 2.000%, **then** contact Engineering to review the calibration and recommend appropriate actions.
- 6.2.34 **If** the “Relative Standard Deviation %” value is less than 2.000%, **then** proceed to Step 6.2.35.
- 6.2.35 Document actual “Relative Standard Deviation %” value on form CP4-ER-0019-F01.
- 6.2.36 Click “Save Library” in the “Calibrate” window to open the “Save Library” window.
- 6.2.37 Click “OK” to save the new data files to the calibration library.
- 6.2.38 **When** the prompt to overwrite the existing data appears, **then** click “Yes” to save the new data files to the calibration library.
- 6.2.39 Click “Save” in the “Calibrate” window to save the check standard method.
- 6.2.40 **When** the “Save Method File” opens, **then** click “OK” to overwrite the current “Ck Std.mth” file.
- 6.2.41 **When** the prompt to overwrite the method appears, **then** click “Yes” to save the method to the analyzer.
- 6.2.42 Close the “Calibrate” window.
- 6.2.43 Double click the “Run Method” icon.
- 6.2.44 Double click on the “Ck Std.mth” to run a new check standard method.
- 6.2.45 Allow for analyzer temperatures to stabilize and the check standard method to run.

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- 6.2.46 Close the “Run Method” window.
- 6.2.47 Double click the “Status” icon.
- 6.2.48 Select the “Parameters” tab.
- 6.2.49 Ensure that the “CkStd Multiplier” value is between 0.9 and 1.1.
- 6.2.50 **If** the “CkStd Multiplier” value is less than or equal to (\leq) 0.9 or greater than or equal to (\geq) 1.1, **then** perform the following:
 - A. Contact Front Line Manager (FLM) and Engineering to review the calibration and recommend appropriate actions.
 - B. **If** Engineering determines the analyzer can **NOT** perform its intended function, **then** notify the FLM of OOT condition according to CP3-SM-0049, *Installed Plant Instrumentation Measuring and Test Equipment*.
- 6.2.51 **If** the “CkStd Multiplier” value is between 0.9 and 1.1, **then** document actual value in the as-left field on form CP4-ER-0019-F01.
- 6.2.52 Proceed to Section 6.6 to return analyzer to normal sampling method **or** return to section(s) 6.3 or 6.7 if performing these sections.

6.3 TCE On-Line Analyzer Analytical Method Calibration

NOTE:

The check standard method calibration (Section 6.2) should be completed prior to performing steps in this section.

Instrument Technician

- 6.3.1 **If** the analyzer is performing a sample run, **then** wait until it is complete and the instrument is in the “Sleeping” mode.
- 6.3.2 **When** the instrument is in the “Sleeping” mode, **then** take the instrument out of automatic operation by pressing the “Esc” key on the analyzer.
- 6.3.3 Connect laptop to analyzer utilizing either the Ethernet or Wi-Fi connection.
- 6.3.4 Close valve HV-052 to isolate the process water supply to the analyzer.
- 6.3.5 Place container under water sampling vessel on the bottom of the analyzer.
- 6.3.6 Open drain valve on the bottom of the water sampling vessel to drain contents.
- 6.3.7 Discard collected water in the C-612 facility sump.

NOTES:

- Calibration solution must be run within six hours of preparation to ensure sample integrity.
- **When** calibration solution is **NOT** in use, **then** containers are to be kept CLOSED.

- 6.3.8 Prepare a 5 parts per billion (ppb) calibration solution by the method given in Section 6.4 for As Found Concentration Check.
- 6.3.9 Inspect water calibration vessel for foreign material or debris.
- 6.3.10 Rinse water calibration vessel with Deionized (DI) water, as needed, to remove foreign material/debris.

CAUTION:

Overfilling the calibration vessel can cause water to be drawn into the analyzer when sample methods are being run, resulting in damage to the analyzer.

- 6.3.11 Pour the calibration solution in the dedicated 2,000 milliliter (mL) water calibration vessel until the level of the solution reaches the line on the container.
- 6.3.12 Remove the water sampling vessel from the analyzer by removing the four wing nuts found on the bottom of the analyzer.
- 6.3.13 Attach the 2,000 mL water calibration vessel to the bottom of the analyzer.
- 6.3.14 Open CMS IQ software.
- 6.3.15 Double click “Run Method” icon.
- 6.3.16 Select the “Water Purge TCE.mth” file **and** click OK.
- 6.3.17 Allow for analyzer temperatures to stabilize and the sample method to run.
- 6.3.18 After sample method is complete, select the “Overlay” button on the CMS IQ toolbar.
- 6.3.19 Open the latest data file for Water Purge TCE sample method to open the data file chromatograph.
- 6.3.20 Ensure that the file name for the data file is highlighted in the “Control Panel” **and** select the “View Search Results” button on the CMS IQ toolbar.
- 6.3.21 Record the measured TCE concentration as the as-found concentration for the analytical method calibration on form CP4-ER-0019-F01.
- 6.3.22 **If** the measured TCE concentration is within the acceptable tolerance of the analyzer, between 3.5 and 6.5 ppb, **then** perform the following:
- A. Record the as-found concentration in the as-left concentration field.
 - B. Proceed to Step 6.3.64.

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- 6.3.23** If the measured TCE concentration is less than (<) 3.0 ppb or greater than (>) 7.0 ppb, **then** prior to proceeding with analytical method calibration, notify the NW/NE Pump and Treat Project Manager.
- 6.3.24** Return to the CMS IQ main screen, **and** click on the “Run Method” icon.
- 6.3.25** Select the “TCE Calibration Sequence.xmth” method sequence from the “Select Method to Run” window, **and** choose “OK.”

NOTE:

The “TCE Calibration Sequence.xmth” file runs four sample methods by varying concentrations using different concentration fill times. The following identifies the sample method file name, concentration fill time, and sample concentration that will be produced during this sample method:

- #1. “Water Purge TCE 06s.mth” – 6 second fill time resulting in a 0.5 ppb concentration
- #2. “Water Purge TCE 36s.mth” – 36 second fill time resulting in a 3.0 ppb concentration.
- #3. “Water Purge TCE 72s.mth” – 72 second fill time resulting in a 6.0 ppb concentration.
- #4. “Water Purge TCE 120s.mth” – 120 second fill time resulting in a 10.0 ppb concentration.

- 6.3.26** **When** the four sample methods are complete, **then** select the “Calibrate” button on the tool bar.
- 6.3.27** Choose the method you would like to calibrate, “Water Purge TCE.mth.”
- 6.3.28** Select the first data file found in the “Data Files” subsection.
- 6.3.29** Press “Delete” on the keyboard until all data files have been removed.
- 6.3.30** Once the old data files have been removed, click on the “Browse” button in the “Data Files” subsection to open the “Select Data Files” window.
- 6.3.31** Select the most recent data file as indicated by the timestamp for the “Water Purge TCE 06s” method.
- 6.3.32** **When** the “Methods Differ” window opens for the data file, **then** click “Yes” to acknowledge this message.
- 6.3.33** **When** the “Inconsistent Concentrations” warning appears, **then** click “OK” to acknowledge this warning.
- 6.3.34** Repeat Steps **6.3.30** through **6.3.33** for “Water Purge TCE 36s,” Water Purge TCE 72s,” and “Water Purge TCE 120s” data files.
- 6.3.35** Select the “Conc/Factor” field (may appear as “Conc/Fa...”) for first data file found in the “Data Files” subsection.

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- 6.3.36** Enter the following values in the “Conc/Factor” field for corresponding data file:
- Water Purge TCE 06s – 0.5
 - Water Purge TCE 36s – 3.0
 - Water Purge TCE 72s – 6.0
 - Water Purge TCE 120s – 10.0
- 6.3.37** Check the checkbox next to each data file found in the “Select” field (may appear as “Sel...”).
- 6.3.38** Check the box next to “Reset Library” to remove old calibration data from the calibration library.
- 6.3.39** Click “Start” to recalibrate the “Water Purge TCE” method.
- 6.3.40** Ensure the value shown in the “Relative Standard Deviation %” is less than or equal to (\leq) 10%.

NOTE:

Engineering may direct that Steps **6.3.1** through **6.3.40** be repeated to recalibrate the analytical standard method or another corrective action.

- 6.3.41** **If** the relative standard deviation for TCE is greater than ($>$) 10%, **then** contact Engineering to review the calibration and recommend appropriate actions.
- 6.3.42** **If** Engineering determines the analyzer cannot perform its intended function, **then** make notifications according to CP3-SM-0049.
- 6.3.43** **If** the relative standard deviation for TCE is less than or equal to (\leq) 10%, **then** proceed to Step **6.3.44**.
- 6.3.44** Click “Save Library” in the “Calibrate” window to open the “Save Library” window.
- 6.3.45** Click “Ok” to save the new data files to the calibration library.
- 6.3.46** **When** the prompt to overwrite the existing data appears, **then** click “Yes” to save the method to the analyzer.
- 6.3.47** Click “Save” in the “Calibrate” window to save the Analytical Method.
- 6.3.48** **When** the “Save Method File” window opens, **then** click “Ok” to overwrite the current “Water Purge TCE” method file.
- 6.3.49** **When** the prompt to overwrite the method appears, **then** click “Yes” to save the method to the analyzer.
- 6.3.50** Remove water calibration vessel from the analyzer **and** pour calibration solution in to the C-612 facility sump.

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- 6.3.51 Generate new 5 ppb calibration solution according to Section 6.4 for As Left Concentration Check.
- 6.3.52 Pour the calibration solution in the dedicated 2,000 mL water calibration vessel until the level of the solution reaches the line on the container.
- 6.3.53 Attach the 2,000 mL water calibration vessel to the bottom of the analyzer.
- 6.3.54 Open CMS IQ software.
- 6.3.55 Double click “Run Method” icon.
- 6.3.56 Select the “Water Purge TCE.mth” file **and** click OK.
- 6.3.57 Allow for analyzer temperatures to stabilize and the sample method to run.
- 6.3.58 After sample method is complete, select the “Overlay” button on the CMS IQ toolbar.
- 6.3.59 Open the latest data file for Water Purge TCE sample method to open the data file chromatograph.
- 6.3.60 Ensure that the file name for the data file is highlighted in the “Control Panel” **and** select the “View Search Results” button on the CMS IQ toolbar.
- 6.3.61 Record the measured TCE concentration as the as-left concentration for the analytical method calibration on form CP4-ER-0019-F01.

NOTE:

Engineering may direct that Steps 6.3.1 through 6.3.40 be repeated to recalibrate the analytical standard method or another corrective action.

- 6.3.62 **If** the measured TCE concentration is less than (<) 3.5 ppb **or** greater than (>) 6.5 ppb, **then** contact Engineering to review the calibration and recommend appropriate actions.
- 6.3.63 **If** the measured TCE concentration is within the acceptable tolerance of the analyzer, between 3.5 and 6.5 ppb, **then** proceed to Step 6.3.64.
- 6.3.64 Remove the water calibration vessel from the analyzer **and** pour calibration solution into the C-612 facility sump.
- 6.3.65 Reinstall the water sampling vessel on to the bottom of the analyzer.
- 6.3.66 Open valve HV-052 to place analyzer back in to service.
- 6.3.67 Proceed to Section 6.6 to return analyzer to normal sampling method.

6.4 Mixing Calibration Solutions

NOTE:

Steps 6.4.1 through 6.4.15 are for mixing a 5 ppb TCE calibration solution.

Instrument Technician

6.4.1 Obtain a dedicated laboratory syringe, volumetric flask, and a minimum of 2,000 mL of DI water.

6.4.2 Fill the volumetric flask with 2,000 mL of the DI water.

NOTE:

Varying concentrations of stock TCE standards can be utilized to generate the calibration solution.

6.4.3 Determine concentration of the stock TCE standard that will be used for the calibration process.

6.4.4 **If** stock TCE standard concentration is greater than (>) 10 parts per million (ppm), **then** prior to proceeding with this activity, consult the Industrial Hygiene (IH) department for additional hazard controls that may be required.

6.4.5 Retrieve a stock TCE standard from the storage freezer.

6.4.6 Check expiration date on the stock TCE standard **and if** stock TCE standard is past the expiration date, **then** do **NOT** use.

6.4.7 **If** the stock TCE standard is within three weeks of the expiration date, **then** prior to proceeding, notify the NW/NE Pump and Treat Project Manager.

6.4.8 Calculate volume of TCE standard aliquot(s) that is required to generate a 5 ppb calibration solution by using formula found on CP4-ER-0019-F01 **and** log the volume on CP4-ER-0019-F01.

6.4.9 Remove label from stock TCE standard vial **and** affix to form CP4-ER-0019-F01 in the “As Found” and/or “As Left” Concentration used blocks.

6.4.10 Carefully open the glass vial by breaking the top off away from your face.

6.4.11 Draw appropriate aliquot(s) of the stock TCE standard from the top of the vial with the syringe **and** carefully add to the volumetric flask to generate 5 ppb calibration solution.

6.4.12 Seal volumetric flask **and** gently swirl to mix solution.

6.4.13 **If** disposing of the remaining calibration solution or stock TCE standard, **then** discard in the C-612 facility sump **and** allow the standard vial to air dry.

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- 6.4.14 Obtain a copy of the manufacturer's Certificate of Analysis **and** maintain copy with the work package.

Operations Personnel

- 6.4.15 File original Certificate of Analysis from the manufacturer in a fireproof file cabinet.

6.5 Daily Data Collection

Operations Personnel

- 6.5.1 **If** the analyzer is performing a sample analysis, **then** wait until the run is complete before reviewing the data.
- 6.5.2 Choose the "Overlay Button" in the CMS Windows Application, **and** choose the method you would like to review.
- 6.5.3 Highlight the data files you would like to review **and** press "OK."
- 6.5.4 Review the chromatograms **and** select the data files with the largest TCE peak.
- 6.5.5 Find the concentration of the largest TCE peak by highlighting the file name **and** choose the "Water Purge TCE.mth" on the menu bar.
- 6.5.6 Record the highest concentration of TCE since the last data collected for a regular sample according to CP4-ER-0017, *Northwest/Northeast (NW/NE) Plume Daily Operational Data Collection and Maintenance*.
- 6.5.7 Close the window with the TCE concentration.
- 6.5.8 **If** the TCE On-Line Analyzer is **NOT** in the automatic mode, **then** go to Section 6.6.

6.6 Restart of the TCE On-Line Analyzer

Instrument Technician or Operations Personnel

- 6.6.1 Choose "Run Method" from the "Operation" menu.
- 6.6.2 Select "Constant Monitoring.xmlth" in the select method window.

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6.7 Performing Calibration Checks and Alarm Testing

NOTE:

Routine calibration checks and alarm testing to verify analyzer performance can be accomplished utilizing the same 25 ppb solution.

Instrument Technician

- 6.7.1 **If** the analyzer is performing a sample run, **then** wait until it is complete and the instrument is in the “Sleeping” mode.
- 6.7.2 **When** the instrument is in the “Sleeping” mode, **then** take the instrument out of automatic operation by pressing the “Esc” key on the analyzer.
- 6.7.3 Connect laptop to analyzer utilizing either the Ethernet or Wi-Fi connection.
- 6.7.4 Open CMS IQ software.
- 6.7.5 Select the analyzer icon located at the bottom of the page to enter analyzer system setup.
- 6.7.6 Double click the “Status” icon.
- 6.7.7 Select the “Parameters” tab.
- 6.7.8 Document current “CkStd Multiplier” value in the as-found field on form CP4-ER-0019-F01.
- 6.7.9 **If** the “CkStd Multiplier” value is between 0.9 and 1.1, **then** document actual value in the as-left field on form CP4-ER-0019-F01 **and** proceed to Step 6.7.11.
- 6.7.10 **If** the “CkStd Multiplier” value is less than or equal to (\leq) 0.9 or greater than or equal to (\geq) 1.1, **then** notify the NW/NE Pump and Treat Project Manager **and** complete Section 6.3 prior to proceeding with calibration checks and alarm testing.
- 6.7.11 Obtain a dedicated laboratory syringe, volumetric flask, and a minimum of 2,000 mL of DI water.
- 6.7.12 Fill the volumetric flask with 2,000 mL of the DI water.

NOTE:

Varying concentrations of stock TCE standards can be utilized to generate the calibration solution.

- 6.7.13 Determine concentration of the stock TCE standard that will be used for the calibration process.
- 6.7.14 **If** stock TCE standard concentration is greater than ($>$) 10 ppm, **then** prior to proceeding with this activity, consult the IH department for additional hazard controls that may be required.
- 6.7.15 Retrieve a stock TCE standard from the storage freezer.
- 6.7.16 Check expiration date on the stock TCE standard **and if** stock TCE standard is past the expiration date, **then** do **NOT** use.

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- 6.7.17** If the stock TCE standard is within three weeks of the expiration date, **then** notify the NW/NE Pump and Treat Project Manager prior to proceeding.

NOTE:

The following equation is to be used to determine the volume of aliquot needed to generate a 2,000 mL calibration solution with a 25 ppb TCE concentration:

$$V_{\text{aliquot}} = \frac{25 \text{ ppb} \cdot 2 \text{ liters}}{c_{\text{standard}}}$$

Where,

Valiquot = Volume of aliquot, in milliliters (1 mL = 1,000 µl)

cstandard = Concentration of stock TCE standard, in ppm (1 ppm = 1 µg/mL)

- 6.7.18** Calculate volume of TCE standard aliquot(s) that is required to generate a 25 ppb calibration solution.
- 6.7.19** Document TCE standard manufacturer, lot number, volume of standard, concentration of stock TCE standard, expiration date, volume of calibration solution, and volume of aliquot used on CP4-ER-0019-F02, *NWP Groundwater System TCE On-Line Analyzer Calibration Check*.

Operations Personnel

- 6.7.20** File original Certificate of Analysis from the manufacturer in a fireproof file cabinet.

Instrument Technician

- 6.7.21** Carefully open the glass vial by breaking the top off away from your face.

NOTES:

- Calibration solution must be run within six hours of preparation to ensure sample integrity.
- **When** calibration solution is **NOT** in use, **then** containers are to be kept closed.

- 6.7.22** Draw appropriate aliquot(s) of the stock TCE standard from the top of the vial with the syringe **and** carefully add to the volumetric flask to generate 25 ppb calibration solution.
- 6.7.23** Seal volumetric flask and gently swirl to mix solution.
- 6.7.24** **If** disposing of the remaining calibration solution or stock TCE standard, **then** discard in the C-612 facility sump **and** allow the empty standard vial to air dry.
- 6.7.25** **If** the analyzer is performing a sample run, **then** wait until it is complete and the instrument is in the “Sleeping” mode.
- 6.7.26** **When** the instrument is in the “Sleeping” mode, **then** take the instrument out of automatic operation by pressing the “Esc” key on the analyzer.
- 6.7.27** Connect laptop to analyzer utilizing either the Ethernet or Wi-Fi connection.
- 6.7.28** Close valve HV-052 to isolate the process water supply to the analyzer.

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- 6.7.29 Place container under water sampling vessel on the bottom of the analyzer.
- 6.7.30 Open drain valve on the bottom of the water sampling vessel to drain contents.
- 6.7.31 Discard collected water in the C-612 facility sump.
- 6.7.32 Inspect water calibration vessel for foreign material/debris.
- 6.7.33 Rinse water calibration vessel with DI water, as needed, to remove foreign material/debris.

CAUTION:

Overfilling the calibration vessel can cause water to be drawn in to the analyzer when sample methods are being run, resulting in damage to the analyzer.

- 6.7.34 Pour the calibration solution in the dedicated 2000 mL water calibration vessel until the level of the solution reaches the line on the container.
- 6.7.35 Remove the water sampling vessel from the analyzer by removing the four wing nuts found on the bottom of the analyzer.
- 6.7.36 Attach the 2,000 mL water calibration vessel to the bottom of the analyzer.
- 6.7.37 Open CMS IQ software.
- 6.7.38 Double click “Run Method” icon.
- 6.7.39 Select the “Water Purge TCE.mth” file **and** click OK.
- 6.7.40 Allow for analyzer temperatures to stabilize and the sample method to run.
- 6.7.41 After sample method is complete, select the “Overlay” button on the CMS IQ toolbar.
- 6.7.42 Open the latest data file for Water Purge TCE sample method to open the data file chromatograph.
- 6.7.43 Ensure that the file name for the data file is highlighted in the “Control Panel” **and** select the “View Search Results” button on the CMS IQ toolbar.
- 6.7.44 **If** on line acceptance criteria are **NOT** met, **then** perform Section 6.3
- 6.7.45 Repeat Steps 6.7.1 through 6.7.44 once the calibration has been completed.
- 6.7.46 Document results and requisite information regarding calibration solution generation on form CP4-ER-0019-F02, for the Analytical Method Calibration Check.
- 6.7.47 Review C-612 Panel View for alarm generation identifying high TCE concentration in system effluent and that the autodialer calls out.

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NOTE:

The alarm can **NOT** be reset until constant monitoring has begun.

6.7.48 Notify the NW/NE Pump and Treat Project Manager to reset the alarm.

6.7.49 Proceed to Section **6.6** to return analyzer to normal sampling method.

7.0 ACCEPTANCE CRITERIA

Acceptance criteria are contained in action steps and data sheets.

8.0 POST PERFORMANCE WORK ACTIVITIES

Operations Personnel

Record all operational activities according to CP4-ER-0017.

9.0 RECORDS

9.1 Records Generated

The following records may be generated by this procedure:

- CP4-ER-0019-F01, *NWP Groundwater System TCE On-Line Analyzer Calibration*
- CP4-ER-0019-F02, *NWP Groundwater System TCE On-Line Analyzer Calibration Check*

9.2 Records Disposition

The records are to be maintained according to CP3-RD-0010, *Records Management Process*.

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Appendix A – Acronyms/Definitions

ACRONYMS

ALWCD – Activity Level Work Control Document

CMS – Continuous Monitoring System

D&R – Deactivation & Remediation

DI - Deionized

DOE – Department of Energy

FLM – Front Line Manager

IH – Industrial Hygiene

JHA – Job Hazard Analysis

mL – milliliter

NW/NE – Northwest and Northeast

OOT – Out of Tolerance

PGDP – Paducah Gaseous Diffusion Plant

ppb – parts per billion

ppm – parts per million

psig – pounds per square inch gauge

SDS – Safety Data Sheet

TCE – Trichloroethene

DEFINITIONS

Operations Personnel – The person performing the technical aspects of this procedure. The person performing this work could have job functions including but **NOT** limited to Facility Manager for C-612 or C-613, the Frontline Supervisor, the NW/NE Pump and Treat Project Manager, or the Operator/Maintenance Mechanic.

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CP4-ER-0019-F01 - NWP Groundwater System TCE On-Line Analyzer Calibration

Analyzer Manufacturer:		Calibration Date:		
Analyzer Model Number:		Technician:		
Analyzer Serial Number:		Badge Number:		
Check Standard Method Calibration				
As-Found Check Standard Multiplier		In Tolerance? <input type="checkbox"/> Yes <input type="checkbox"/> No	Acceptable As-Found Check Standard Multiplier Range	0.7 - 1.3
As-Left Check Standard Multiplier		In Tolerance? <input type="checkbox"/> Yes <input type="checkbox"/> No	Acceptable As-Left Check Standard Multiplier Range	0.9 - 1.1
Relative Standard Deviation (RSD%)	%	In Tolerance? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	Acceptable As-Left Relative Standard Deviation	< 2.0%
Comments:				
Analytical Method Calibration				
Calculate Volume of Aliquot Needed to Generate 2 Liter Solution with a 5 ppb TCE Concentration:				
As-Found		As-Left		
$V_{\text{aliquot}} = \frac{5 \text{ ppb} \cdot 2 \text{ liters}}{C_{\text{standard}}} = \text{ ______ } \text{ ml}$		$V_{\text{aliquot}} = \frac{5 \text{ ppb} \cdot 2 \text{ liters}}{C_{\text{standard}}} = \text{ ______ } \text{ ml}$		
Where, V_{aliquot} = Volume of aliquot, in milliliters (1 ml = 1,000 μ l) C_{standard} = Concentration of TCE standard, in ppm (1 ppm = 1 μ g/ml)				
Analytical Standard Used for As-Found Data and Calibration (affix label and attach copy of COA):		Analytical Standard Used for As-Left Data (affix label and attach copy of COA):		
As-Found Concentration:	ppb	In Tolerance? <input type="checkbox"/> Yes <input type="checkbox"/> No	Acceptable As-Found Concentration Range	3.0 - 7.0 ppb
As-Left Concentration:	ppb	In Tolerance? <input type="checkbox"/> Yes <input type="checkbox"/> No	Acceptable As-Left Concentration Range	3.5 - 6.5 ppb
Comments:				
Calibration Approvals				
<hr/> Technician Performing Calibration (Print/Sign/Date)		<hr/> NW/NE Pump and Treat Project Manager or Designee (Print/Sign/Date)		

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CP4-ER-0019-F02 - NWP Groundwater System TCE On-Line Analyzer Calibration Check

Analyzer Manufacturer:	Date:
Analyzer Model Number:	Technician:
Analyzer Serial Number:	Badge Number:
Analytical Method Calibration Check	
TCE concentration for calibration check solution to be generated:	ppb
Volume of aliquot used to generate calibration check solution:	ml
Volume of calibration solution	ml
Analytical Standard Used for Analytical Method Calibration Check (affix label and attach copy of COA):	
Analyzer Measured TCE Concentration:	ppb
Note: Analytical Method Calibration Check will be considered acceptable if the analyzer measures a TCE concentration is within $\pm 30\%$ of the TCE concentration for the calibration check solution (e.g. acceptable range for a 25 ppb calibration check solution is 17.5 - 32.5 ppb).	
Comments:	
Analytical Method Calibration Check Approvals	
_____ Technician Performing Calibration Check (Print/Sign/Date)	_____ NW/NE Pump and Treat Project Manager or Designee (Print/Sign/Date)