Appendix N

Deviations from the Work Plan

Appendix N

2024 Clean Harbors PFAS Tests
Deviations from the Work Plan

by
William L. Troxler, P.E.
Focus Environmental, Inc.
Knoxville, Tennessee 37909

Table of Contents

Abbreviations

ATG	Alliance Technical Group
DRE	destruction and removal efficiency
EPA	U.S. Environmental Protection Agency
FTIR	Fourier transform infrared
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
OTM	Other Test Method
RFA	request for analysis
COC	chain of custody

1 Summary of Deviations

This section summarizes deviations from the Work Plan. For changes that were technical in nature, this section provides an assessment of the impact of the deviation on data quality.

1. Table 2-1. Runs were incorrectly numbered in Table 2-1 in the Test Plan. The correct sequence of planned run numbers should have been as shown in Table 2-3:

    - 11-12-24: Run 1A, Run 1B, Run 1C
    - 11-13-24: Run 2A, Run 2B, Run 2C
    - 11-14-24: Run 3A, Run 3B, Run 3C.

2. Table 2-1. Because of delays in starting testing on November 12, 2024, there was not sufficient time in the workday for Clean Harbors Environmental Services and Alliance Technical Group (ATG) staff to complete Run 1A, 1B, and 1C. The project team decided to postpone Run 1A until November 13, 2024. The schedule for completing the remaining runs was modified to complete all nine test runs in the originally planned 3 day period. The change in run sequence had no effect on data quality.

3. Table 2-1. The flow controller on the Eurofins Other Test Method (OTM)-50 Silonite canister did not function properly during Run 1B because of the low atmospheric pressure at the plant (elevation 4,300 feet above mean sea level) and high vacuum (approximately 15 inches water column) in the stack. The U.S. Environmental Protection Agency (EPA) OTM-50 Silonite canister was equipped with a critical orifice which did function correctly. The initial Run 1B was renamed Run 1B-1 and this run number was applied only to the EPA Silonite canister. Another run was performed and named Run 1B-2 using the Eurofins OTM-50 Silonite canister equipped with the EPA critical orifice. These changes had a minor effect on data quality, except that the EPA and Eurofins canisters were not collected simultaneously and cannot be considered as duplicates.

4. Table 2-1. Run 1B-1 and Run 1B-2 were completed by ATG staff. Because of Department of Transportation work hour restrictions, ATG personnel had to leave the site before Run 1C could be conducted. The Run 1C sampling was performed by one of the authors of OTM-50, William Roberson (EPA), and Dr. William Anderson (Focus). Because the Alliance staff were not on site during Run 1C, EPA Method 2 and EPA Method 4 were not performed to collect stack gas flow data. Run 1C was performed immediately after Run 1B-2. Therefore, stack flow data from Run 1B-2 was used as surrogate data for Run 1C. . Estimated PFAS emission rates for Run 1-C could be biased either slightly high or low, depending on the actual stack gas flow rate during Run 1C compared to the measured stack gas flow rate during Run 1B-2. Using surrogate flow rate data for Run 1C likely had a minimal effect on data because the measured gas flow rates from other runs where OTM-50 samples were collected (Runs 2B, 2C, 3B, and 3C) ranged from 31,878 dscfm to 32,687 dscfm. However, during Run 1-C, concentrations of all analytes in both the Eurofins OTM-50 and the EPA OTM-50 canister were non-detect. Therefore, stack emission rates were extremely low for Run 1-C even if there is a small degree of uncertainty regarding the stack gas flow rate used to calculate the emissions. Therefore, substituting the Runs 1-B-2 stack gas flow data for Run 1-C had a minimal effect on data quality.

5. Table 6-1. Added units (% opacity) to Baghouse Broken Bag Detectors. Added CEMs monitoring

data (O_2, CO_2, CO, SO_2, NO_x, THC).

6. Table 8-1. Added estimated fluorine mass flow rate from PFAS spiking calculation (1.02 lb/hour).

7. Table 9-1. Added calculation of destruction and removal efficiency (DRE) value that could be determined for C_2F_6 using the Fourier Transform Infrared (FTIR) instrument minimum detectable concentration.

8. Table 9-1. During Run 1C, C_2F_6 was spiked at 12.41 lb/hr (Table 6-1) versus the rate in Table 9-1 of the Work Plan (12.43 lb/hour). FTIR monitoring results for C_2F_6 during Run 1-C indicated the C_2F_6 concentration in the stack gas was near non-dect. The project team also determined that shakedown testing time for C_2F_6 injection could be reduced from four hours to less than one hour and the pre-spiking time before a run could be reduced from one hour to less than one minute. Since the total amount of pre-test spiking time could be reduced, more C_2F_6 was available for spiking during test Run 2C and 3C than had originally been planned. The test team members (Focus, EPA, and SSI, and Spectrum) decided to increase the C_2F_6 spiking rate during Runs 2C and 3C to attempt to elicit a measurable response for C_2F_6 on the FTIR instrument. Therefore, the C_2F_6 feed rate was increased to approximately 45 lb/hour for Run 2C and Run 3C. Increasing the C_2F_6 spiking rate had a positive effect on data quality since it allowed demonstrating a higher DRE value.

9. Figure 9-1. After Run 1C was completed, the valving and tubing on the outlet side of the C_2F_6 gas cylinder were noted to be collecting frost on their surfaces. Before conducting Run 2C, a blanket heater was installed on the C_2F_6 gas cylinder to keep the gas temperature at approximately 100°F. The valves and tubing between the C_2F_6 cylinder and the mass flow meter were also heat traced to keep them from freezing. This change had a positive effect on data quality by allowing C_2F_6 flow rate to be controlled more accurately.

10. Table 11-2. Historical data for “At Std Cond, dscfm” was corrected for Runs 7, 8, and 9 and for the Average, Minimum, and Maximum values. This correction was communicated to Alliance during the pre-test meeting.

11. Table 12-1. The test run sequence was revised as previously described in Item 3.

12. Table 12-2. The daily test schedules varied from day to day based on which runs had previously been conducted. See the ATG Test Report, Appendix H, Tables 2-1 through 2-4 for the actual date and start and stop times for each test run.

13. During Run 3A, the direct burner tanker pump which was feeding AFFF failed at 10:12. ATG was notified to cease sampling at 10:14. This time coincided with a sampling port change. ATG resumed sampling when the pump was placed back online at 10:40. Conducting stack sampling for two minutes while the pump was offline could have biased emissions approximately one percent low. The one percent value was estimated as the time that ATG was sampling when AFFF was offline (2 minutes) divided by the total sampling time for the OTM-45 train (192 minutes) x 100 percent.

14. Appendix H. Master Sample List. Sample S-2045 is mis-coded as 2045 (S-1 is missing). No effect on data quality, sample was coded in correctly on receipt at laboratory.

15. Appendix H. Master Sample List. Samples are missing for OTM-50 Silco lined canisters for Run

1B, 1C, 2B, 2C, 3B and 3C. The Master Sample List was updated to include these samples. The original RFA/COC form showed these samples, but they did not have a unique four digit sample number. The samples numbers were added to the RFA/COC form after sample receipt at the Eurofins Knoxville Laboratory.

16. Appendix H. Master Sample List. The spray dryer solids stream was not homogeneous and contained a mixture of dust and slag materials. EPA requested Eurofins to evaluate methods to produce a homogeneous sample for analysis. The plan that was developed after the test included sieving the material into three size fractions, grinding each fraction, analyzing each fraction separately, and calculating mass weighted average concentrations of analytes.

17. As noted in Item number 16 above, due to heterogeneity in the spray dryer solid matrix, all three primary spray dryer solid samples and associated QA/QC samples were marked as "Hold at Lab" on The Master Sample List and RFA/COC. Additionally, three , 1-gallon sample containers of Spray Dryer Solids from runs 1A, 2A, and 3A were submitted for additional fractionation and analysis pending EPA guidance. These samples were added to The Master Sample List with sequential numbers 1053, 1054, and 1055, respectively, and marked as "Hold at Lab" on the request for analysis/chain of custody.

18. Appendix H. Master Sample List. Fuel oil was not fed through the combined fuel oil/used motor oil lance, therefore fuel oil process samples were not collected. Also, the sludge port was used to spike C_2F_6 therefore no sludge process sample was collected. The Master Sample List was updated to strike out those samples in Run 1A, 2A, and 3A.

19. For quality assurance samples collected during Run 1A, extra sample volume was collected for matrix spikes (MS) and matrix spike duplicates (MSDs) (one MS and one MSD bottle) from all feed streams, utilities, and residual streams, except for the drum and tote Educt stations. These additional QA samples were added as individual lines to The Master Sample List and annotated with "-MS" and "-MSD" to the Sampling Coding ID. At the request of EPA, duplicate samples were collected from all feed streams, utilities, and residual streams (excluding the Educt drum & Educt tote). These additional duplicates were submitted to Eurofins and marked "hold" on the RFA/COC. The duplicates were inserted in The Master Sample List with a "D" identifier added to the Sequential No. and "-DUP" annotated to the Sampling Coding ID.

20. Two samples (1051 and 1052) were added to The Master Sample List based on observations made during the field event. Sample 1051 was collected as a rinsate blank from a new lot of the HDPE funnels used to transfer composite samples to laboratory bottleware in the on-site laboratory. Sample 1052 was a sample of a new PTFE baghouse filter.

21. RFA/COC Forms. During the handoff of stack gas samples from Chris McBride (Focus) to Doug Cahill (Eurofins), the sample transfer date was entered as 11/15/24. It should have been entered as 11/16/24. The error in recording the handoff date was discovered when the samples were delivered to the Eurofins Knoxville laboratory. Dr. William Anderson discussed the error in recording the dates with the Eurofins Knoxville Laboratory sample coding custodian. The dates on the RFA/COC forms were corrected and initialized by Mr. McBride (on 11/21/24) and by Mr. Cahill (on 11/26/24).