

# **Exhibit A**

Prepared by PTaylor&Associates, LLC

## **Final Report: Assessment of a Report on PFAS Destruction Testing Results at Clean Harbors' Aragonite, Utah Hazardous Waste Incinerator**

Prepared for:

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## Outline of Assessment

This assessment is divided into two sections. The first section gives an overall assessment of the PFAS Destruction Testing Results. The focus is the attainment of the primary and secondary objectives of the testing program. As stated in the PFAS Destruction Testing Results report, the primary objective was to demonstrate high DRE for the four spiked chemicals PFOA, PFOS, PFHxS, and HFPO-DA. The secondary objectives were to develop a mass balance for incidental PFAS in the waste infeed and to demonstrate high HF removal efficiency coincident with the high PFAS DRE. Some additional comments are provided at the end of this section. The second section is a detailed review of the PFAS Destruction Testing Results report and Appendices with specific questions, answers from Clean Harbors or its representatives, and in some instances, follow-up questions and answers. This approach laid the foundation for the overall assessment of the testing program.

## Overall Assessment

The primary objective of demonstrating high DRE for spiked PFAS compounds was clearly achieved. Although there were serious issues with the transfer of the stack gas samples to the analytical laboratories, the analytical data are not in question given the extremely high thermal stability of PFAS at ambient or near-ambient conditions. Calculations are provided in Attachment A demonstrating the extreme thermal stability of PFOA and PFOS, corroborating the assertions of the report that the stack gas sample transfers to the analytical laboratory did not affect the accuracy of the measurements.

Greater than 99.9999% DRE was achieved for the spiked PFAS (PFOA, PFOS, PFHxS, and HFPO-DA) for average temperatures of greater than 1000 C in both the RK and the AB and residence times of greater than 3.0 sec (AB). While the testing stands on its own merit, there are recent bench-scale experimental (PFOS) and theoretical (*ab initio* modeling) published studies (PFOS, PFBA, and PFPA) that provide scientific support for these measurements. I have taken the kinetics reported in the published studies and calculated DREs and the data is shown in Attachment B. The calculations suggest temperatures of 800 C is sufficient to achieve > 99.9999 DRE of these compounds for afterburner residence times of 2.0 sec. (Note that the highlighted cells point to the lowest energy pathways for each substance.) It is interesting to note that the dominant breakdown pathway for PFOS, PFBA, and PFPA is not C-C scission along the C<sub>8</sub> carbon chain, but instead HF elimination from the polar end of the molecules. The C<sub>8</sub> chain is left intact and requires higher temperatures for the C-C bonds to be ruptured. This is the likely source of the C<sub>1</sub> and C<sub>2</sub> perfluorocarbon (PFC) PICs that are of concern.

The secondary objectives were considered the most difficult to achieve as they involve more complex sampling issues.

The PFAS mass balance is credible and the resulting assertions regarding a conservative DRE are also credible. It is possible that some of the PFAS that was not sampled in the infeed may have been more difficult to gasify and burn, contrary to the assertions in the report. However, studies have shown that PFOA is very reactive on surfaces at low temperatures. And once in the gas-phase, PFOA and other PFAS appear to be of modest thermal stability. Based on current understanding, I believe that the PFAS DRE is conservative as stated in the report.

The HF removal efficiency assertion is not quite as clearcut. The data collected in Attachment C, the revised fluorine mass balance calculations provided by Focus Environmental, point to serious issues with respect to the fluoride measurements. While a HF removal efficiency of >99.6% is reported, the sinks for the HF were not accurately quantitated leading to some question about the validity of the reported HF removal efficiency. A confounding issue is the claim that all fluorine in the waste is converted to HF upon combustion. For this assumption to be scientifically valid, a large excess of hydrogen to fluorine is needed. This data is not provided in the report. Likely sources of hydrogen beyond what is in the waste feed would be an auxiliary fuel, e.g., natural gas, burned during the testing and/or the presence of moisture in the waste feed. Proof of complete conversion of the organic fluorine to HF can also be shown with the analytical data. However, this was not the case. There was a demonstrated lack of accuracy of the analytical methods for measuring inorganic fluorine in the residual streams. The data in the revised fluorine mass balance (Attachment C) indicate mass balances of only 2-10%. An assessment of the reasons for this low recovery of fluorine suggests there were likely analytical issues associated with the slag and spray dryer solids samples. Development of better analysis methods (for complex matrices) for inorganic fluorine are needed before high HF removal efficiencies can be reported with confidence at the full-scale.

In a related matter, it is also likely from the analysis of the ratio of fluorine in the brine relative to the waste infeed that the accuracy of the organic fluorine measurements is also questionable. This ratio varied over a very wide range (52 to 457%) in this testing program.

In summary, development of better analysis methods for both organic and inorganic fluorine are needed to support PFAS performance testing at the full-scale.

### **Additional Comments**

PIC Formation – Full-scale testing is not generally a good source of data to examine PIC formation. In this testing, the low DRE of PFBA in runs 1, 3 and 7 (< 99%) is suggestive of PIC formation as there were many longer chain perfluoroalkyl acids in the waste infeed that could yield PFBA by a simple mechanism, C-C bond rupture. However, the higher DREs for PFBA in runs 4-6 where PFOA was spiked at higher concentrations is inconsistent with this hypothesis. In addition, the low PFBA DREs may simply be due to very low waste infeed concentrations.

PFBA has also been seen in high relative concentrations in the stack gases for other PFAS testing programs, e.g., MacGregor et al., 38<sup>th</sup> IT3 Conference, January 27, 2021. From Attachment B, it is shown that PFBA is not predicted to be thermally stable relative to other PFAS such as PFOS and PFPA. It is therefore unclear what mechanism is responsible for its relatively high emission rate compared to other PFAS. Perhaps it is not related to PIC formation but some other phenomena or a sampling and analysis artifact.

Final Comments – It is my opinion that the high PFAS DREs observed in this testing program are consistent with the state of the science of PFAS combustion. This science suggests that many PFAS compounds including the ones spiked in the waste infeed in this program are of modest thermal stability. The larger question from an environmental viewpoint is the complete mineralization of these substances and the prevention of emission of highly stable C<sub>1</sub>- C<sub>2</sub> PFCs. The extensive fluoride measurements performed in this study are commendable although they did not provide accurate data in support of the mineralization of these substances. It should also be noted that demonstration of PFAS mineralization was not a goal of this testing program.

## References to the Literature used for Calculations in Attachment 1 and 2

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M. Altarawneh, M. H. Almatarneh, and B. Z. Dlugogorski, "Thermal Decomposition of Perfluorinated Carboxylic Acids: Kinetic Model and Theoretical Requirements for PFAS Incineration," *Chemosphere*, 286, 2022, pp. 131685.

S. W. Benson, "Thermochemistry and Kinetics of Sulfur-Containing Molecules and Radicals," *Chemical Reviews*, 78, 1978, pp. 23.

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W. Tsang, D. R. Burgess, Jr., and V. Babushok, "On the Incinerability of Highly Fluorinated Organic Compounds, *Combustion Science and Technology* 139, 1998, pp. 385.

N. H. Weber, S. P. Stockenhuber, C. S. Delva, A. A. Fara, C. C. Grimison, J. A. Lucas, J. C. Mackie, M. Stockenhuber, and E. M. Kennedy, "Kinetics of Decomposition of PFOS Relevant to Thermal Decomposition Remediation of Soils, *Ind. Eng. Chem. Res.* 60, 2021, pp. 9080.

## Specific Observations from Review of Report on PFAS Destruction Testing Results at Clean Harbors' Aragonite, Utah Hazardous Waste Incinerator

### Executive Summary:

1. From report: It should also be noted that high temperature treatment processes such as incineration probably have an effect on hidden PFAS mass in a waste stream similar to the laboratory chemical/thermal oxidation in the TOPA.

Question: Please provide further context and justification for this assertion.

Response: The TOPA process is a chemical oxidation process at mildly elevated temperatures. The chemical oxidation cleaves some bonds in the PFAS molecules, resulting in lower molecular weight compounds that are more likely to be target analytes. For example, AFFF may contain a variety of PFAS compounds, including 4:2 FTS, 6:2 FTS, 8:2 FTS, 10:2 FTS, 12:2 FTS. The first four compounds are target analytes by EPA Method 537, whereas 12:2 FTS is not. The TOPA analysis revealed increases in the concentrations of the first four compounds, which were likely the result of the oxidation of higher molecular weight compounds, including 12:2 FTS or possibly other compounds. It is likely that a similar process could occur in high temperature thermal treatment processes, but at a much higher conversion efficiency.

Follow-up response: No additional questions.

2. From report: Due to the high efficiency of the dual dry scrubber/wet scrubber system, low HF emissions were measured throughout the testing. Measured values of fluorine ion in the stack gas samples were all J flagged values (values falling between the method detection limit and the limit of quantitation). The associated HF stack emission rate averaged 8.13E-03 lb/hr during the testing and the HF gas stack concentrations ranged from 0.07 to 0.14 parts per million dry volume basis, corrected to 7% oxygen. There are currently no HF emission limits under either the RCRA Hazardous Waste Incinerator Standards or the HWC Maximum Achievable Control Technology Standard. However, as points of comparison, the RCRA hydrogen chloride emission limit is 4.0 lb/hr and the Maximum Achievable Control Technology hydrogen chloride limit for existing HWC is 32 parts per million dry volume basis, corrected to 7% oxygen. HF is a Hazardous Air Pollutant (HAP) under the Clean Air Act, with a major source threshold of 10 tons/year. At the average HF emission rate measured during testing, annual emissions would be less than 100 pounds/year.
3. Question: Is there any evidence to suggest complete destruction of the spiked PFAS compounds occurred?

Response: There is a significant difference between "destruction" and "mineralization". Per

the RCRA definition of destruction, a molecule is destroyed if it is chemically altered. This test program conclusively showed that >99.9999% destruction and removal efficiency could be obtained for a number of PFAS compounds if there was sufficient material in the feed to perform this demonstration, based on the analytical detection limit in the stack gas.

“Mineralization” requires showing that feed compounds are converted to stable end products including CO<sub>2</sub> and HF. Demonstrating complete PFAS mineralization would require performing a fluorine balance and verifying that all organic fluorine was converted to HF. This was not a goal of this program for the following reasons:

- 1) The plant was run under “normal” operating conditions, which included feeding solid waste (containers and shredded materials). Due to heterogeneity of these materials, there was no way to collect representative samples and analyze them for fluorine. Therefore, the total fluorine input to the system is not quantifiable for the solid waste materials. Hence, a complete fluorine balance is impossible for this type of system when feeding normal waste feed materials.
- 2) There may be multiple sources of fluorine in the feed material in addition to PFAS compounds. Examples include pesticides, pharmaceuticals, etc. There is no practical way to distinguish between the sources of fluorine in the feed materials that contribute to the HF produced in the offgas.

Follow-up response: No additional questions.

#### Section 1:

4. From report: PFAS are particularly challenging to degrade, both environmentally and thermally, due to the strength of the multiple carbon-fluorine (C-F) chemical bonds in these compounds.

Question / Response: There is growing evidence that the destruction of the C-4 through C-10 perfluorocarboxylates and C4-C10 perfluorosulfonates can be achieved at temperatures well below 1000 C. The destruction of lighter C1-C4 fluorocarbons derived as PICs from these parent compounds are likely to require higher temperatures, perhaps above 1000 C for CH<sub>x</sub>F<sub>y</sub> where x = 0 or 1 and y = 3 or 4, for complete destruction to CO<sub>2</sub> and HF.

Response: No question asked, Clean Harbors agrees with the observation.

Follow-up response: None

5. From report: During the testing, the operating temperature of the RK ranged from 1,893 to 2,008 degrees Fahrenheit (F) and the operating temperature of the ABC ranged from 2,052 to 2,110 F.

Question: In Appendix B, minimum temperatures ranging from 1731 F (Test Condition 1) to

1773 F (Test Condition 3) were recorded for the RK. Are you reporting average temperatures in the statement from the report?

**Response:** The values cited in the text are the range of average temperatures for the nine test runs. They are not the absolute minimum and maximum values recorded in Appendix B.

**Follow-up response:** It is suggested that the text be modified as described in the response.

**Response 2:** Clean Harbors agrees that if the report is revised this statistical basis for the temperatures should be clarified.

6. From report: Combustion gas residence time in the ABC is estimated at 2 to 3 seconds.

**Question:** Are there calculations to back up this statement? I did not see any calculations in the report or the Appendices.

**Response:** Afterburner residence time cannot be measured directly; it can only be calculated from mass and energy balance calculations. Residence time is also not a regulatory parameter, therefore, there are no calculations of afterburner residence time in the report. However, based on mass and energy balance calculations that were performed for other purposes using the PFAS test data, the estimated average gas residence time in the afterburner during the PFAS tests was 3.1 seconds.

**Follow-up response:** No additional questions.

## Section 2:

7. From report: This secondary test program goal was to demonstrate, to the extent practicable, an overall mass balance for the process and to calculate DREs for as many of the 49 target PFAS analytes as possible, based on PFAS feed rates and stack gas analytical detection limits.

**Question:** What mass balance are you referring to? The mass of PFAS compounds being fed? It is unclear on what is met by mass balance here. Mass balance can have several different meanings based on the context used.

**Response:** "Mass Balance" in the context of this report refers to calculating the mass of each individual PFAS compound in each process stream, including waste feed streams, reagent input streams, and process residual streams to the extent possible. The response to Question #3 explained the limitations on sampling and analysis of solid material, hence, PFAS materials in the solid waste feeds could not be included in the mass balance. Figures 6-1, 6-2, and 6-3 present a summary of the total PFAS mass balance data for each type of stream that was sampled and analyzed.

Please note that there are caveats that should be applied to these data because of how analytical detection limits were handled in calculations. For waste feed, reagent, and process residual streams, non-detects were assigned a value of zero in all calculations. For stack gas samples, all non-detects were assigned the detection limit in all calculations. This was done so that DRE calculations were done on the most conservative basis possible (i.e., likely omitting some PFAS mass in the feed and likely overestimating PFAS stack gas emission rates). Therefore, reported DRE values are likely underestimated.

Follow-up response: The wording here describing mass balance within the context of this report should be considered in a future revision of the report.

Response 2: Clean Harbors agrees that if the report is revised the use of the term “mass balance” should be clarified.

8. From report: Due to the high molar proportion of fluorine in PFAS chemicals, thermal treatment of these materials breaks the C-F bonds and generates byproduct HF, a HAP regulated under the Clean Air Act. Maximizing thermal breakdown of PFAS into products of complete combustion also maximizes the production of HF. Hence, current U.S. Environmental Protection Agency (EPA) guidance on PFAS thermal treatment emphasizes the importance of not only high PFAS DRE but also achieving low stack gas concentrations of HF. Stack gas samples for quantifying the presence of HF were withdrawn using an EPA Method 26A sampling train during each of the nine test runs to measure HF emission rates.

Question: Was PFAS destruction to HF measured directly in this study? This would be a direct and clear way to demonstrate a F mass balance and complete destruction of the PFAS in the feed.

Response: See response to Question #3.

Follow-up response: No additional questions.

### Section 3:

9. From report: A detailed work plan was developed before conducting the tests to facilitate systematic execution of the field activities, including sampling, analysis, and quality control, to ensure that the project objectives would be met. Tests were run under each of three different process conditions typical of normal waste processing operations. Test Condition 1 (Test Runs 1-3) was intended to establish a baseline for results without adding additional PFAS spiking compounds to the waste feed. During Test Condition 2 (Test Runs 4-6), the feed rates of PFOA, PFOS, PFHxS, and HFPO-DA were augmented by spiking to facilitate calculation of DRE values for these compounds. During Test Condition 3 (Test Runs 7-9), AFFF concentrate was also fed to the incinerator.

Question: What was the purpose of test condition 3, how does it relate to test condition 1 and 2, and how was the data used to demonstrate PFAS DRE?

Response: The purpose of Test Condition #1 was to feed normal waste feed materials that were not known to specifically contain PFAS compounds (baseline conditions). The purpose of Test Condition #2 was to feed normal waste feed materials (similar to Test Condition #1) but to also spike four PFAS compounds at feed rates that had been calculated for each compound to be sufficient to demonstrate >99.9999% DRE based on the estimated analytical detection limit in the stack gas for each specific spiking compound. The purpose of Test Condition #3 was to feed a waste material (AFFF) that was believed to contain a significant concentration of PFAS and is also a commercial product that is likely to be processed through the incinerator on a routine basis.

DRE values were calculated for every target analyte for every run for each test condition. However, it should be noted that feed concentrations of many of the PFAS target analytes were too low to demonstrate the 99.99% DRE value required by RCRA regulations at the analytical detection limits that were achievable in the stack gas.

Follow-up response: No additional questions.

10. From report: The mass balance for the four spiked PFAS also included the spiked amounts.

Question: What is meant by this statement?

Response: For the overall mass balances as shown in Figures 6-1, 6-2, and 6-2 the amounts of PFAS spiked during Test Condition #2 make up a significant fraction of the overall PFAS mass fed to the system. Please see Appendix A, page 5, table titled "Total PFAS Input – Contribution from Waste Feeds and Spiked Materials". For Runs 4, 5 and 6, the spiked materials make up 83, 76, and 92% respectively of the total mass of PFAS in the feed materials. This must be considered in comparing mass balance results between Test Conditions #1, #2 and #3.

Follow-up response: No additional questions.

11. From report: Table 3-1 presents the approach for calculating spiking rates for the four PFAS compounds to demonstrate a DRE > 99.9999% for each of the four spiked PFAS compounds. Demonstrating DRE > 99.9999% is equivalent to demonstrating that for every million mass units of a POHC introduced into an incinerator, only one mass unit is detected at the stack. Hence, it is necessary to work backwards from the achievable detection limit for the specific analyte in the stack gas. A sample calculation demonstrating the contribution of the spiking component inputs to the DRE calculation are presented in Table 3-1. Note that the example calculation presented below is actually based on achieving > 99.99995% DRE. This contingency factor is necessary to

account for uncertainty in the PFAS method detection limit (MDL) and stack gas flow rate.

Question: What is the accuracy of the PFAS feed rates and stack emission rates? To how many significant digits?

Response:

One key factor that affects the accuracy of the calculations of PFAS feed rates and stack emissions is how non-detect analytical values were handled in calculations. Please see response to Comment #7 for a discussion of this issue. As explained in that comment, the most conservative approach was taken to handling non-detects in performing DRE calculations.

Mass feed rates of waste materials are reported in units of lb/hr to five or six significant figures (i.e., XXX.XX or X,XXX.XX). Concentrations of PFAS in liquid feed materials are reported in concentrations of ng/l to three significant figures (X.XX). Stack gas sampling train fractions (4) are reported in units of ng/sample to four significant figures (X.XXX). Stack gas sample volumes are reported in units of dscf to five significant figures (XXX.XX). Stack gas flow volumes rates are reported in units of dscfm to five significant figures (XX,XXX).

Follow-up response: Given the accuracy stated above, are the DRE numbers stated in the report still correct? In other words, the report states the spiked PFAS was destroyed to a DRE greater than 99.9999%. Is this still correct given the low feed rates and the accuracy of the measurements?

Response 2: The calculations were performed following the protocol described in RCRA for hazardous waste incineration (40 CFR 264.343) and are believed to be correct. An error propagation test based on number of significant figures has not been performed nor is such a test required by RCRA.

It should be noted that part of the test planning included calculating the mass of each PFAS compound that was required to be spiked to demonstrate >99.9999% DRE based on the analytical detection limit in the stack gas. As noted in Table 6-2, the mass of PFAS in the waste feed materials was insufficient to demonstrate >99.9999% DRE for many of the PFAS target analytes.

12. From report: All of the temperatures recorded for samples on receipt at the laboratory were higher than 4 degrees Celsius (C). Interviews with EAL employees revealed that proper temperature measurement procedures were not observed. Further, it was confirmed through analysis of XAD media blank samples retained at the lab and **the fact that PFAS are very thermally, chemically, and biologically stable**, that XAD sample integrity was likely not compromised by temperatures that were above the target value. Other Test Method 45 (OTM-45) does not require refrigeration of the sample filter, which indicates there is a low level of concern with sample loss at ambient temperatures. Details of this reconciliation are summarized in a Technical Memorandum

provided in Appendix E.

Question: What is the basis for this statement (highlighted text)? I will provide calculations to test this assertion.

Response: Comment acknowledged, and Clean Harbors concurs that PFAS compounds do not have unusual thermal stability at typical combustion temperatures. However, the statement is made in the context of the temperatures that were recorded for various samples, which ranged from ~8 to ~24°C. The statement is believed to be true in that PFAS compounds would be thermally stable within this temperature range, but no data have been found to confirm this.

Follow-up response: Calculations are provided that give support for this assertion based on the prior work of Krusic et al, 2005.

Response 2: Based on telecon with Dr. Taylor, I understand that these calculations will be provided in a separate report that he is preparing and were not intended to be part of this document.

13. From report: The Test Plan was based on a comprehensive approach to sampling that included as many process inputs and outputs as practicable. The purpose of this was to develop as complete a PFAS mass balance as possible and to identify potential sources of PFAS entering the process and PFAS sinks for materials leaving the process. Sampling included waste feed streams, process treatment chemical inputs, process residue streams, and stack gas. The principal exclusions from sampling were the containerized, shredded and bulk solids waste feed streams. Due to their complexity and heterogeneity, it was considered infeasible to sample and perform PFAS analyses on these materials. Hence, any PFAS compounds contained in these waste streams were not included in the mass balance or DRE calculations.

Question / Comment: Once again, not sure what is meant by mass balance here.

Response: See response to Question No. 7.

Follow-up response: No additional questions.

14. From report: Field QC for PFAS in process samples included rinsate/equipment blanks and field blanks collected per the master sample matrix (Appendix G), and field duplicates collected at a rate of 10% of the total process samples. At the laboratory, for every extraction batch ( $\leq 20$  samples), a method blank and a matrix spike were performed. Surrogates were spiked into the samples to verify extraction efficiency and internal standards were spiked into extracts to verify instrument drift. The Gas Chromatography-Liquid Chromatography instrument was calibrated using a minimum of five concentration levels. All assays were bracketed by passing continuing calibration verification standards. A maximum of 10 samples were assayed between continuing

calibration verifications. All laboratory QC procedures and results are documented in the EAL Report.

Question: Is this correct (highlighted text)? My understanding is that LC/MS/MS was used to analyze for PFAS.

Response: The analytical instrument was described incorrectly in the text, it was in fact LC/MS/MS.

Follow-up response: No additional questions.

15. From report: EPA Method 3A is an instrumental test method that was used to measure the concentration of O<sub>2</sub> and CO<sub>2</sub> in the stack gas, and nitrogen by difference. Three effluent gas samples were collected in Tedlar bags during each run. Following the completion of the test run, the contents of the bags were conveyed to continuous emissions analyzers that measured the concentration of O<sub>2</sub> and CO<sub>2</sub>. The average of the three bags for each run was used for the O<sub>2</sub> and CO<sub>2</sub> concentration. The performance requirements of the method were met to validate data.

Question: Was CO measured in any of the test runs? PFAS are within a class of known flame inhibitors and may result in CO formation, depending on the concentrations fed. It would be good to verify that the PFAS had no effect on the combustion process.

Response: CO is continuously monitored and recorded as a condition of the plant's RCRA permit. The permit limit is 100 ppmv, dry basis, 1 hour rolling average. CO emissions were well within permit limits during all test runs. However, the CO emission concentrations are not reported within the PFAS report.

As reported in Appendix A, page 2, PFAS Mass Flow rate, the average total PFAS feed rate in all feed streams was 0.0132 lb/hr (excluding PFAS spiking chemicals). The average total PFAS spiking rate during Runs 4-6 was 0.348 lb/hr. Data in Appendix B shows that the average total waste feed rate during all runs was 12,818 lb/hr. Therefore, PFAS (without spiking) make up only 0.00001% of the total waste feed material. Including the spiking compounds, PFAS made up only 0.003% of the total waste feed. Clean Harbors believes it is unlikely that PFAS at these concentrations would have a significant effect as a flame inhibitor. However, Clean Harbors is not aware of any qualitative studies to support this assumption.

Follow-up response: No additional questions.

16. From report: Figure 3-1 and Table 3-2.

Question: Was the tubing within the OTM-45 sampling train rinsed and analyzed for PFAS? This includes all tubing from the particulate filter to the 5th impinger as shown in Fig. 3.1.

Response: Stack testing was conducted according to the procedures in OTM-45, which requires rinsing all sampling train glassware, including the sampling probe, impingers, and all connecting glassware.

Follow-up response: No additional questions.

#### Section 4:

17. From report: The laboratory performed data validation by comparing the final data deliverable/report to the project objectives, summarizing QC outliers in the final deliverable, and applying data validation qualifiers to associated results. The data were evaluated against project data quality objectives and measurement performance criteria, such as precision, accuracy, and completeness, as shown in Table 4-1.

Question: Was third-party data validation conducted?

Response: Third party data validation was conducted for all analytical data. This Data Validation Report is presented as Appendix J of the test program report.

Follow-up response: No additional questions.

#### Section 5:

No questions

#### Section 6:

18. From report: The secondary objectives of developing a mass balance for incidental PFAS in waste infeed and demonstrating high HF removal efficiency coincident with high PFAS DRE were also achieved.

Question / Comment: It is unclear that successful demonstration of the secondary objective was achieved. Specifically, high HF removal efficiency.

Response: HF removal efficiency was calculated using a procedure that is analogous to the procedure required by the RCRA Incineration Regulations to demonstrate HCl removal efficiency. This procedure is based on measuring the amount of organic chlorine in the waste feed materials and converting it to an HCl using a stoichiometric conversion factor (assuming 100% conversion efficiency). The amount of HCl in the stack gas is measured by capturing the chloride ion in impingers and converting it to HCl using a stoichiometric conversion factor. The formula for HCl removal efficiency is:

$$((\text{HCl feed} - \text{HCl stack gas}) / \text{HCl feed}) * 100.$$

Calculated HF removal efficiency values for each test run are presented in Table 6 in the test report. Measured HF removal efficiencies were >99.6% in all cases.

Follow-up response: Is Clean Harbors still confident that the HF was removed to >99.6% given the large variance in the fluoride measurements in the residual streams (Attachment B)?

19. From report: Depending on the Test Condition, between 24 and 26 of the 49 target PFAS analytes were not detectable in the waste infeed and DREs for these could not be calculated. However, stack gas concentrations for all 49 target PFAS analytes were either not detectable, or if detectable, the mass emission rates from the stack were extremely low, generally ranging from  $10^{-9}$  to  $10^{-7}$  lb/hr. PFBA, PFHxA, perfluoropentanesulfonic acid (PFPeS), perfluoro-2-methoxyacetic acid (PFMOAA), N-EtFOSE, N-MeFOSE, and perfluoro(3,5,7,9,11-pentaoxadodecanoic) acid (PFO5DA) had reported emission rates between  $10^{-7}$  and  $10^{-6}$ . PFBA, the compound with the highest calculated emission rate, was present in the method blank (XAD resin media blank). Therefore, the reported PFBA emission rate is likely biased high, and the DRE is biased low for at least some of the test runs by this blank contamination. Calculated stack emission rates for all 49 target PFAS analytes are presented in Table 6-3 and stack emission concentrations are presented in Table 6-4.

Question: What are the blank-corrected emission rates for PFBS in Table 6-3?

Response: See blank corrected emission rates in Attachment D.

Follow-up question: The DRE for PFBA is less than 99% for runs 1, 3, and 7 (Table 6-2). Did the blank-corrected data make a measurement improvement (order of magnitude) in the DRE numbers for runs 1, 5, and 7? Can you offer any explanation for the low DRE besides a low initial concentration in the waste feed?

Response 2: See revised Attachment D which shows blank corrected DRE calculations for PFBA. Blank correction generally results in a small increase in DRE, but far less than an order of magnitude for each run that was blank corrected.

Table 6-2 shows that there are several PFAS target analytes with DRE values <99.99%, even though the analyte was non-detect in the stack gas. This result is clearly caused by low concentrations of the PFAS target analyte in the feed material.

PFBA may also be a PIC. It is the lowest molecular weight (C4) carboxylic acid and would be expected to be a breakdown product of higher molecular weight carboxylic acids. Data presented by MacGregor (attached) also shows PFBA as residual product in multiple residual stream matrices (ash, water, stack gas) which may corroborate the PIC theory.

20. From report: The HF removal efficiency, based on the HF potential to emit and the and

measured HF in the stack gas, averaged 99.7% for the nine test runs.

Question: This statement assumes that you measured all the fluorine in the infeed. I don't think this is true. It also assumes that all fluorine fed into the incinerator was converted to HF. What basis do you have for this second assertion?

Response: Table 6-4 in the project report presents HF removal efficiency data and all values are reported as ">" values partially for the reason noted (fluorine was not sampled and analyzed in the waste feed). There were several reasons for reporting HF removal as a ">" value as explained in footnotes a, b, and c of Table 6-4. The statement in the text should also be qualified as a ">" value.

As described in the response to Question 18, HF removal efficiency was calculated is procedures analogous to those described in the RCRA incineration regulations for calculating HCl removal efficiency, which assumes that all chlorine in the feed material is converted to HCl. The point is acknowledged that there is a possibility that some fluorine could be converted to compounds other than HF. However, fluorinated products of incomplete combustion were not measured during this test program.

Follow-up response: I agree with the response. It is possible if not likely that some of the input F was converted to stable PICs such as CF<sub>4</sub> and C<sub>2</sub>F<sub>6</sub> in this testing. The report should reflect this current status of the science with respect to complete F mineralization.

Response 2: See initial response to Question #3.

21. From report: Table 6-5.

Question: How is Intermediate HF defined? A footnote should be added to this table providing that calculation. HF removal rate is calculated by subtracting HF stack emission rate from fluorine from waste feed with the difference divided by the fluorine from waste, correct? If so, what is the purpose of adding the Intermediate HF column? What does this data signify?

Response: HF removal percentage is calculated by converting all fluorine in the feed to HF and converting all fluorine in the stack gas to HF and then performing the calculation as describe above. "Intermediate HF" is the potential HF calculated by converting all of the fluorine in the waste feeds and spiking chemical to HF by using a stoichiometric equation (as shown in the table heading below the term "Intermediate HF". Clean Harbors agrees that the use of the term "Intermediate HF" is potentially confusing, and the term should be removed from the table.

Follow-up response: No additional questions.

22. From report: The technical memorandum also addresses the recorded temperatures of

the sample coolers upon receipt at the laboratory, which ranged from 6 °C to 13.3 °C except for one measurement of 19.4 °C recorded for three coolers containing non-hazardous stack gas samples. However, it is believed that this measurement was not made in accordance with standard procedures, as documented in the memorandum (Appendix E). The measured temperatures for the three coolers containing non-hazardous stack gas samples were noted as an exceedance relative to standard requirements, and associated sample data were qualified as estimated (J or UJ) per standard validation procedures. Additionally, the hazardous stack gas samples required shipment in specialized hazardous materials compliant fiberboard boxes, which precluded cooling during shipping; the temperatures of these samples upon delivery were greater than 23 °C.

Question: This data (highlighted text) is missing from Appendix J. Data for the non-hazardous waste samples is provided twice.

Response: Please confirm that the question references the proper Appendix. Temperature data for hazardous gas constituents is provided in Appendix E, Reconciliation of Sample Handling Deviations, pages 41-42 (Fed Ex documents).

Follow-up Response: In the current version of Appendix E, Attachment 2 (COCs for hazardous stack gas samples) is a duplicate of Attachment 3 (COCs for non-hazardous stack gas samples). Appendix E should be corrected with the appropriate COCs for Attachment 2.

Response: The term “Hazardous” refers to the DOT shipping classification of the samples, not the toxicological properties of the samples. The COCs in question refer to the OTM-45 samples. The “front-half rinse”, “back-half rinse”, and “impinger rinse” fractions are a 95% methanol/5% NH<sub>4</sub>OH solvent mixture that are defined as hazardous materials for DOT shipping purposes. These remaining four samples (front half filter, 1<sup>st</sup> XAD trap, Condensate, Breakthrough XAD) are considered non-hazardous per DOT regulations. These groups of samples should have been listed on separate COC forms and shipped separately. However, they were erroneously combined on a single COC form. This form is included in the referenced Attachment 2 (Hazardous Stack Gas Samples COCs) and Attachment 3 (non-hazardous Stack Gas Samples COCs) because the form contains information for samples in both hazardous and non-hazardous categories.

23. From report: Although the impact of temperature on the stability of PFAS in samples similar to those collected for this project has not been specifically documented, PFAS compounds are generally highly stable and resistant to degradation under normal environmental conditions. The stability of these compounds is reflected in Method OTM-45, which does not require refrigeration of sample filters. Therefore, although the data were qualified based on the temperature exceedances, the exceedances do not affect data usability.

Question: A calculation will be provided to test this assertion (highlighted text) using kinetic

data from the literature.

**Response:** See response to Question #12.

**Follow-up response:** Calculations are provided that give support for this assertion based on the prior work of Krusic et al, 2005.

**Response 2:** See response to Question 12.

24. From report: The media blanks associated with the OTM-45 train were prepared and analyzed as method blanks. These blanks were collected in association with the XAD resin, the impinger solution, and the filter. The two blanks associated with the impinger solution were non-detect for all analytes. Analytical results for the filter blank were non-detect except for PFTeDA, which had a concentration of 0.061 ng/sample. Analytical results for the two XAD resin blanks were non-detect for all analytes except PFBA, PFHxA, and PFMOAA. **The detected result for PFBA was 5.53 ng/sample**, the detected results for PFHxA were 0.247 ng/sample and 0.234 ng/sample, and the detected results for PFMOAA were 1.35 ng/sample and 1.39 ng/sample.

**Question:** Is this consistent with footnote (d) in Table 6-4? The footnote seems to suggest that PFBA was detected in different components of the OTM-45 sampling train for different runs. It's confusing.

**Response:** See data in Attachment A. Footnote (d) refers to the concentration of PFBA in the Method Blank. The XAD-resin reagent blank was used as a method blank. Footnote (d) should be reworded as follows:

**(d) Detected as a contaminant (5.53 ng/sample) in method blank No. MB-12020-PFAS. The XAD-2 resin reagent blank was used as the method blank. The PFBA mass in the following stack gas sample fractions exceeded the mass in the method blank:**

- Run 1 – Back-half**
- Run 5 – Back-half**
- Run 5 – Breakthrough XAD Resin**
- Run 7 – Back-half**

**Follow-up response:** No additional questions.

## Section 7:

25. From report: The results of testing conducted at Clean Harbors' Aragonite Incineration Facility from 17 to 19 June 2021 successfully demonstrate that the process can effectively destroy legacy terminal PFAAs such as PFOA, PFOS, and PFHxS, and the Gen-X

PFAS HFPO-DA, at DREs exceeding 99.9999%, when the feed rate of these materials is augmented and DREs are calculated using very conservative assumptions. It is worth noting that this is the required performance level for thermal destruction of polychlorinated biphenyls and dioxins/furans, which are demonstrably more hazardous and generally less thermally stable than currently (federally) unregulated PFAS.

Comment: The current, evolving scientific understanding does not support this statement. PFAS parent compounds are almost certainly less thermally stable than PCBs.

Response: Comment acknowledged. Clean Harbors is not aware of any published studies comparing the thermal stability of PFAS compounds with PCBs that were conducted using identical or similar test procedures. Therefore, this statement in the original report is unsubstantiated by any published data that is known to Clean Harbors and should be removed.

Follow-up response: No additional questions.

26. From Report: Section 7.2 Title: Incidental PFAS DRE and Mass Balance

Comment: Title is misleading. PFAS mass balance (as fluorine) was not reported. No evidence for the complete destruction of the spiked PFAS was provided.

Response: See responses to Question #3 and Question #7. Demonstrating a PFAS mass balance as fluorine was not a goal of the testing program and was technically impracticable for the reasons noted in the previously referenced responses.

Follow-up response: No additional questions.

27. From report: Mass balance results indicate that total PFAS input into the system in the treatment chemicals and process water are extremely low, with non-detect or close to non-detect concentrations of all analytes in the process water, TMT, and powdered activated carbon. The soda ash solution has a PFAS input into the system in the range of 10<sup>-6</sup> to 10<sup>-5</sup> lb/hr. This mass was comprised exclusively of FBSA, which was detected in all nine runs.

Mass balance results indicate the total PFAS emitted from the system in the slag, spray dryer solids, baghouse dust, and stack gas are extremely low (10<sup>-8</sup> to 10<sup>-4</sup> lb/hr for each stream). Mass emission rates in the slag and stack gas are approximately equal and the mass emission rates in the spray dryer solids and baghouse dust are both about one order of magnitude less than the flow rates in the slag or stack gas.

Comment: The significance of these paragraphs is unclear to me. Are you saying there was little background PFAS in the incoming streams and unit operations (except for AFFF and the spiked PFAS in test condition 2) and little PFAS in the stack gas and air pollution control

systems? I agree with the latter assertion but not so sure about the first; the report states that not all of the incoming streams were analyzed for PFAS or total organic fluorine.

**Response:** Comment is correct in that PFAS could not be analyzed in solids streams as described in response to Comment #3. The text should be qualified to clarify that it is based only on those streams that were sampled and analyzed and does not address any PFAS that may be in solid waste streams which were not sampled and analyzed.

**Follow-up response:** No additional questions.

28. From report: Given that laboratory standards enabling targeted analysis exist for only about 50 of the thousands of extant PFAS, other analytical tools such as **non-targeted PFAS analysis and Total Organic Fluorine, combined with TOPA**, could be employed in the future to more completely characterize the PFAS profiles in the waste and other process streams, as well as in the stack gas.

**Comment:** This statement (highlighted text) relates to both determination of complete PFAS destruction and the ability to perform a meaningful F balance. The need for this analytical capability could be further emphasized as such outcomes would be beneficial for both your industry and the general public.

**Response:** Not a question, comment acknowledged about analytical capability. However, as explained in the response to Comment #3, Clean Harbors does not believe it is possible to perform a meaningful fluorine balance on a commercial hazardous waste incinerator feeding a normal mixture of solid materials for the reasons previously noted.

**Follow-up response:** None.

29. From Report: Performing a fluorine balance, considering all fluorine inputs, could provide insights into how and where fluorine is removed from the system. **Most (probably nearly all) of the organic fluorine is expected to be oxidized to form HF within the RK or ABC.** This HF may then react with inorganic components of the slag, be neutralized and collected in the spray dryer solids, collected in the baghouse as an inorganic solid, or neutralized in the scrubber.

**Question:** What is the basis for this assertion (highlighted text)?

**Response:** The assertion is based on the typical operating temperatures in the rotary kiln and afterburner (~2,000°F) as described in Comment # 5, the gas phase residence time of 3.1 seconds as described in the response to Comment #6, and the oxygen content of the stack gas (~11%) as described in Appendix H of the test report. These operating conditions are believed to be sufficient to convert F to HF at a high efficiency. However, there is no publicly available data that is known to Clean Harbors to quantify the efficiency of the fluorine to HF conversion.

Follow-up response: No additional questions.

## Appendices

### Appendix A:

Fluorine Mass Balance Spreadsheet – The two tables entitled: Fluorine Feed Rate and Fluorine Mass Flow Rate in Residual Streams. Mass Balance Closure (%).

Comment: The mass balance closure for fluorine should be >100% if fluorine input in waste streams to incinerator is not fully characterized as repeatedly asserted in the final report. The data here do not provide strong support or corroboration for that claim as 6 of 9 test runs give only ~ 100% recovery (+/- 10%) and only one test run is well above 100% (130%). I suggest that the assertion in the final report that PFAS DREs are conservative be reconsidered in light of this data.

Response: The data in the Fluorine Mass Balance spreadsheet in Appendix A is incorrect, it is an obsolete version that uses assumed fluorine concentrations in Residual Streams rather than actual measured values. Actual measured values were not available when the spreadsheet was originally developed.

The corrected fluorine mass balance is attached (Attachment C, Fluorine Balance). The calculated fluorine mass balance closures ranged from approximately 2-10%. However, it is believed that the analytical method used to analyze fluorine in the spray dryer ash and baghouse dust was not appropriate for these matrices and therefore returned ND values for most of the samples. There is empirical evidence to support this, in that the mass flow rates of fluorine in the brine ranged from ~1.5 to 9.8 lb/hr. The brine is evaporated in the spray dryer, and this mass of fluorine should have been detected as fluorine in the spray dryer solids or the baghouse dust. It should be noted that there could be additional fluorine in these two streams if fluorine was initially removed from the flue gas as it passed through these two devices (which is highly likely).

A better indication of the fluorine mass balance closure is provided by the ratio of fluorine in the brine to the total fluorine in the feed. These values ranged from 105% to 457% for Runs 1-3 and from 52-65% for Runs 4-9. Ratios exceeding 100% may indicate that there was a significant amount of fluorine in the solid feed materials that could not be sampled and analyzed. This could have resulted in more fluorine being recovered in the brine than could be accounted for in the feed materials that were analyzed.

For Runs 4-9, the ratio of fluorine in the brine to fluorine measured in the feed materials was <100%. This could be the result of fluorine initially being removed from the flue gas as it passed through these spray dryer and baghouse, but not being detected because of analytical issues.

As noted in the response to Comment #3, PFAS DRE values and mineralization efficiency to HF are two totally different parameters. Clean Harbors believes assertions that calculated DRE values are reported on a conservative basis is correct because of how non-detects were used in DRE calculations for waste feed (ND assumed 0.0) and stack gas samples (ND assumed to be present at the ND).

Follow-up response: The large variance in the fluoride measurements in the residual streams suggest some significant analytical issues. This should be reflected in the report and additional testing and/or R&D is recommended to further investigate this issue.

Response: Clean Harbors agrees with this conclusion and acknowledges that there were likely analytical issues associated with the slag and spray dryer solids samples that need to be investigated in future test programs.

#### Appendix E:

Question: The 2<sup>nd</sup> page, last paragraph, states that “The sample temperature issues involved at the recording of 19.4 C of non-hazardous stack gas samples, and temperatures ranging from 23.4 to 24.1 C for hazardous stack gas samples . . .” The COC documents providing the temperatures for the hazardous stack gas samples is not provided in this Appendix.

Response: Temperature data for hazardous gas constituents is provided in Appendix E, Reconciliation of Sample Handling Deviations, pages 41-42 (Fed Ex documents). The shipment of the hazardous gas samples was not properly documented on a COC document.

Follow-up Response: In the current version of Appendix E, Attachment 2 (COCs for hazardous stack gas samples) is a duplicate of Attachment 3 (COCs for non-hazardous stack gas samples). Appendix E should be corrected with the appropriate COCs for Attachment 2.

Response 2: See response to Question 22.