

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

CENTER FOR ENVIRONMENTAL MEASUREMENT AND MODELING RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF RESEARCH AND DEVELOPMENT

December 2, 2019

Mr. Steven E. Flint, Director – Division of Air Resources New York State Department of Environmental Conservation (NYSDEC) 625 Broadway Albany, New York 12233-3250

Dear Mr. Flint:

In response to your July 20, 2018 request for assistance to qualitatively identify per- and polyfluoroalkyl substances (PFAS) in the emissions from an industrial source that is thermally processing polytetrafluoroethylene (PTFE) powders, I am pleased to provide the enclosed report. The report provides qualitative targeted and non-targeted analytical results identifying and tentatively identifying various PFAS and PTFE thermal degradation products from process emission samples. These samples were collected by the U.S. Environmental Protection Agency's (EPA's or the Agency's) Office of Research and Development (ORD) in ongoing collaborative technical support to the New York State Department of Environmental Conservation (NYDEC) at a PTFE powder thermal processing facility.

NYSDEC's request was to aid in the investigation to the presence of PFAS in the environment near manufacturing facilities of interest. This related well to ORD's research capabilities and interests to apply qualitative targeted and non-targeted sampling and analysis methods for discovery of the nature and extent of PFAS environmental occurrence associated with industrial releases. ORD continues to develop emissions sampling and analytical methods for many PFAS compounds in various media, including some of those included in the report.

The report provided does not interpret exposure or risk from the tentative identification of PFAS compounds and PTFE thermal degradation products. EPA does not currently have health-based standards, toxicity factors, or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the report data indicate the presence (or lack) of specific PFAS in the process emissions, we do not have sufficient information to offer interpretations related to human or environmental exposure, or risk.

Thank you for inviting us to be part of this effort to further both EPA's and NYDEC's understanding of this important issue. This is just one of many Agency efforts that demonstrates EPA's commitment to cooperative federalism. If you have any questions or concerns, do not hesitate to contact me at (919) 541-2106 or via email at watkins.tim@epa.gov or Lara Phelps at (919) 541-5544 or via email at phelps.lara@epa.gov. I look forward to our continued work together.

Sincerely,

Timothy H. Watkins

Timothy H Watkins

Director

Enclosure

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Joint ORD/New York State Department of Environmental Conservation: Saint-Gobain PTFE Sintering Facility Emissions Characterization Study

Study Description

The New York State Department of Environmental Conservation (NYSDEC), Division of Air Resources (DAR), and EPA Region 2 have requested the assistance of EPA's Office of Research and Development (ORD) in performing targeted and non-targeted Poly- and Perfluoroalkyl Substances (PFAS) emissions analyses to qualitatively identify air emissions from process source operations. Specifically, ORD has been requested to qualitatively identify various PFAS and other thermal degradation products in stack emissions from the polytetrafluoroethylene (PTFE) process. Quantitative analyses are not within the scope of this study.

The process studied is an industrial source where bulk PTFE (\sim 1,000 lb) is molded and then sintered in an oven. The sintering process involves a gradual heating of the PTFE billet until it reaches the desired sintering temperature (\sim 700 °F). The overall sintering process requires \sim 5 days, including \sim 2 days for cooling. Potential emissions include residual PFAS as well as thermal degradation products, such as PTFE monomers.

The technical objectives of this joint ORD – NYSDEC study were to characterize the emissions, as comprehensively as possible, from the PTFE sintering process. This includes characterizing emissions as a function of sintering process time and temperature using targeted and non-targeted measurement approaches. Investigating the potential presence of PFOA is the primary targeted measurement of interest, as well as the presence of thermal degradation products such as tetrafluoroethylene (TFE), hexafluoropropylene (HFP), and perfluoroisobutylene (PFIB). Non-targeted measurements were conducted to tentatively identify potential additional process emission compounds not identified by targeted measurement approaches.

This data report is intended to provide a simple representation and summary of the study results. Therefore, the description of methods and quality assurance are brief and high-level. In this report, we provide identification and tentative identification of PFAS compounds and PTFE thermal degradation products. We do not interpret exposure or risk from these values. EPA does not currently have health-based standards, toxicity factors, or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the data provided in this report indicate the presence (or lack) of specific PFAS in the process emissions, we do not have sufficient information to offer interpretations related to human or environmental exposure and risk.

ORD personnel were responsible for all on-site sample collection and measurements, as well as all analyses performed. ORD's sampling, analysis, and report team that contributed to this effort are listed in Table 1.

ResponsibilityPersonnelProject Technical LeadJeff RyanEmissions samplingJeff Ryan, Ken Krebs, Theran Riedel, John OffenbergLaboratory chemistryJames McCord, Ingrid GeorgeQuality Assurance ReviewLibby Nessley, Bob WrightManagement coordination and reviewLara PhelpsReport PreparationJeff Ryan, Ken Krebs

Table 1. EPA Office of Research and Development sampling, analysis, and report team.

Methods in Brief

Sampling Approach:

Multiple gaseous emissions samples were collected at the sintering process uncontrolled exhaust. Emissions samples were collected as a function of sintering process time and temperature over the course of ~70h of emissions testing.

Whole air samples were collected with SUMMA® canisters for USEPA Method TO-15 (EPA/625/R-96/010b, January 1999) targeted qualitative analyses using gas chromatography/mass spectrometry (GC/MS), targeted qualitative thermal degradation products analyses (GC/MS), and qualitative non-targeted PFAS screening analyses, also using GC/MS.

An aqueous, four-impinger, midget impinger train was used to capture hydrophilic PFAS for targeted and non-targeted, qualitative PFAS analyses. This approach focuses on the qualitative capture of hydrophilic PFAS compounds, including PFOA, that may be present in the PTFE sintering process exhaust and is not intended to quantitatively capture all potential PFAS compounds.

ORD's iodide adduct high resolution chemical ionization mass spectrometry (CIMS) was used as a real-time, on-line process characterization monitor. The CIMS is capable of detecting specific polar compounds such as polyfluorinated carboxylic acids (PFCAs), including PFOA, as well as fluorotelomer alcohols (FTOHs).

All samples were collected from a sampling manifold where the process emissions are extracted from the stack with a dedicated sampling system. The sampling system, depicted in Figure 1, consists of an unheated sample probe and heated tubing from the probe to the heated manifold. The emissions sample was withdrawn from the stack with a bypass vacuum pump. All emissions samples were collected from the heated manifold positioned on the vacuum side of the sample pump.

A total of 16 - 2h SUMMA® and impinger train emissions samples were collected over the course of the ~70h oven heating cycle. No emissions samples were collected during the oven cooling cycle. A single ambient SUMMA sample was collected indoors in an area in the vicinity of the sintering ovens. Prior to initiation of the oven cycle, a system blank was performed where single SUMMA and impinger samples were collected while nitrogen was introduced at the sample probe and routed through the entire sampling system in the same manner as process emissions as a means to assess potential overall system contamination.

Stack Wall
SS Tubing

Heated
Filter
Orifice
Valve

Summa
To Midget
Impinger Train

Stack Wall

Sypass
Vacuum
Pump

Figure 1. Sampling System

Analytical Approach:

Targeted GC/MS analysis of the SUMMA® canisters for TO-15 VOCs was conducted on a GC coupled to a quadrupole mass spectrometer. Samples were analyzed under electron ionization selective ion monitoring (SIM), MS conditions. Chromatographic 'area under the curve' are reported for each identified compound. No internal standard masses (i.e. 'mass added') nor peak areas are reported, even if added to these samples. As a result, these targeted analyses are qualitatively only. The TO-15 target analyte list is presented in Table 2.

Table 2. TO-15 Target VOCs

Propylene	Methylene Chloride	2-Methylhexane	Bromoform
Propane	3-Chloro-1-Propene	2,3-Dimethylpentane	Styrene
Dichlorodifluoromethane	1,1,2-Trichloro-1,2,2	Tert Amyl Methyl Ether	1,1,2,2-Tetrachloroethane
Chloromethane	Carbon Disulfide	3-Methylhexane	o-Xylene
Isobutane	2,2-Dimethylbutane	1,2-Dichloropropane	Nonane
Dichlorotetrafluoroethane	trans-1,2- Dichloroethene	Bromodichloromethane	Bromofluorobenzene
Vinyl Chloride	Cyclopentane	1,4-Dioxane	Chlorotoluenes
1-Butene	2,3-Dimethylbutane	Trichloroethene	n-Propylbenzene
1,3-Butadiene	1,1-Dichloroethane	Isooctane	m-Ethyltoluene
Butane	Methyl-t-Butyl-Ether	Methyl Methacrylate	1,3,5-Trimethylbenzene
trans-2-butene	Vinyl Acetate	Heptane	1,2,4-Trimethylbenzene
Bromomethane	2-Methylpentane	cis-1,3-Dichloropropene	Tert-Butyl Benzene
cis-2-butene	2-Butanone	4-Methy-2-Pentanone	1-Ethyl-4-Methyl Benzene
Chloroethane	3-Methylpentane	Methylcyclohexane	o-Ethyltoluene
Ethanol	2-Chloroprene	trans-1,3- Dichloropropene	1,3-Dichlorobenzene
Vinyl Bromide	1-Hexene	1,1,2-Trichloroethane	1,4-Dichlorobenzene
Acetonitrile	cis-1,2-Dichloroethene	2,3,4-Trimethylpentane	n-Decane
Acrolein	Diisopropyl ether	Toluene	Sec-Butyl Benzene
Acetone	Ethyl Acetate	2-Methylheptane	1,2,3-Trimethylbenzene
iso-Pentane	n-Hexane	2-Hexanone	1,2-Dichlorobenzene
Trichlorofluoromethane	Chloroform	Dibromochloromethane	o-Cymene
Isopropyl Alcohol	Tetrahydrofuran	3-Methylheptane	1,3-Diethylbenzene
1-Pentene	Ethyl Tert-Butyl Ether	1,2-Dibromoethane	1,2-Diethylbenzene
Acrylonitrile	Methylcyclopentane	Octane	n-Butyl Benzene
n-Pentane	1,2-Dichloroethane	Tetrachloroethene	Undecane
Isoprene	2,4-Dimethylpentane	1,1,1,2-Tetrachloroethane	1,2,4-Trichlorobenzene
trans-2-pentene	1,1,1-Trichloroethane	Chlorobenzene	Naphthalene
cis-2-pentene	Benzene	Ethylbenzene	Dodecane
cis-z-pentene	Belizelle	1	
Tert-Butanol	Carbon Tetrachloride	m-Xylene	Hexachlorobutadiene

Targeted GC/MS analysis of the SUMMA® canisters for TFE, HFP, and PFIB, as well as 4:2, 6:2, and 8:2 telomer alcohols, were conducted on a GC coupled to a quadrupole, time-of-flight mass spectrometer (TOF/MS). Samples were analyzed under electron ionization full-scan, MS conditions. Gas standards for TFE, HFP, and the telomer alcohols were used to aid in compound identification/compound absence. Chromatographic 'area under the curve' are reported for each identified compound. No internal standard masses (i.e. 'mass added'), nor peak areas are reported even if added to these samples. As a result, these targeted analyses are qualitatively only. As no gas standard for PFIB can be used, tentative compound identification/compound absence was determined with the aid of reference spectra and retention indices.

Originally, the intent was to perform both targeted and non-targeted liquid chromatography-mass spectrometry (LC-MS) analyses on the impinger samples. Targeted analyses were not performed and deemed not necessary due to several factors. The LC/MS system used for targeted PFAS analyses was not available during the time period of this study. Since this study is limited to qualitative analyses, targeted PFAS analyses for purposes of quantification were not necessary. The LC/MS used for non-targeted analyses was also more powerful and will detect/identify the targeted PFAS as part of the non-targeted analyses.

The non-targeted PFAS analyses were performed on an ultra-performance liquid chromatograph (UPLC) system, coupled to a high resolution, high mass accuracy mass spectrometer. Where possible, PFAS compounds are positively identified against PFAS analytical standards. However, the availability of PFAS analytical standards is limited and often inadequate to identify the PFAS present in many environmental samples. The primary method for tentative PFAS identification is based on the interpretation of mass spectral data – molecular mass along with patterns of fragmentation.

Spectral information is compared to mass-spectral libraries (such as the *U.S. EPA CompTox Chemicals Dashboard* https://comptox.epa.gov/dashboard) to tentatively identify compounds present. In addition, PFAS compounds are tentatively identified using specialized spectral interpretation software and analyst expertise. PFAS compounds are tentatively identified to various levels of confidence, depending on the level of combined evidence from manual examination of MS/MS fragmentation spectra and/or comparison with mass spectral libraries. This approach is further described in McCord et al. 2019.

The mass spectrometer detector provides integrated peak areas for each compound within the chromatogram. The peak area counts are proportional to the mass of PFAS in the sample. However, without a standard we are not able to derive a mass or concentration value, and results are considered qualitative. As a result, only peak area counts (responses) are reported and these non-targeted analyses are qualitative only.

¹ McCord, J., Strynar, M. Identifying Per- and Polyfluorinated Chemical Species with a Combined Targeted and Non-Targeted-Screening High-Resolution Mass Spectrometry Workflow. *J. Vis. Exp.* (146), e59142, doi:10.3791/59142 (2019). https://www.jove.com/video/59142/identifying-per-polyfluorinated-chemical-species-with-combined

Quality Assurance/Quality Control Summary

This study adhered to the associated Quality Assurance Project Plan (QAPP) (Joint ORD/New York State Department of Environmental Conservation: Saint-Gobain PTFE Sintering Facility Emissions Characterization Study QA Track ID G-AEMD-0031868-QP-1-0) prepared and approved prior to testing. The only deviation from the original study plan was that targeted PFAS analyses were not performed on the impinger samples. Relying solely on the non-targeted PFAS analyses does not limit reported data quality in any way. The justification for this has been described above.

The collection and analysis of a variety of reagent and method blanks to account for any PFAS contamination that may have occurred during sampling and analysis was integral to the interpretation and validation of reported data. Multiple blank samples were collected and analyzed, including trip, field, laboratory, instrument, and system blanks. The primary purpose of these blanks is to isolate what is attributable to the samples due to contamination, and to ensure that the data reported are based on actual sintering process emissions. All blank measurements are reported along with emissions data.

The compounds identified and reported are based on their relative relationship to the various blanks and their measured levels. As the primary means of data validation, only those compounds with area counts/responses ≥ 10 times the associated blank level are considered as present in process emissions. There are a total of 16 emissions samples. A compound is considered present if it is found at ≥ 10 times the associated blank level for even a single sample. However, area counts for all 16 samples are reported. The peak area data for determining these relative ratios are contained in this report.

Summary of Results

The qualitative SUMMA[®] results for the targeted TO-15 analyses are reported in Table 3. The compounds identified and the associated area counts are reported. A total of 39 of the 119 VOC compounds targeted for analysis were present in process emissions. The \geq 10 times the associated blank level is relative to the nitrogen system blank sample.

The qualitative SUMMA® results for the targeted TFE, HFP, PFIB, and the 4:2, 6:2, 8:2 telomer alcohol analyses are reported in Table 4. The compounds identified and the associated area counts are reported. Only TFE and 4:2 telomer alcohol were present in process emissions. HFP and PFIB were not present in process emissions. The ≥ 10 times the associated blank level is relative to the nitrogen system blank sample.

The CIMS was operated continuously from the start of process oven heating cycle until the end of the heating cycle, when the cooling cycle began. One-minute data collection averages were electronically stored. The CIMS responses over the entire heating cycle are graphically depicted in Figure 2. Only the C2 (perfluoroethanoic acid), C3 (perfluoropropanoic acid), and C4 (perfluorobutanoic acid) perfluorocarboxylic acids (PFCAs) were present in process emissions. It should be noted that the relative responses to each other should not be interpreted as concentrations relative to each other, since the absolute CIMS response varies among individual compounds.

The impinger results for the non-targeted PFAS analyses are presented in Tables 5-7. The compounds tentatively identified and the associated area counts are reported. A total of 15

polyfluoro PFAS compounds were tentatively identified. The presence of these PFAS compounds in process emissions was limited to sample 1 only. This indicates that these compounds were emitted very early in the oven heating process. The ≥ 10 times the associated blank level is relative to the nitrogen system blank sample. With the exception of the tentative identification of (3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-Heptadecafluorodecyl) hydrogen maleate and 6:2 fluorotelomer phosphate diester (which were diagnostically matched against the U.S. EPA CompTox Chemicals Dashboard), only the molecular mass and formulae can be established. Actual PFAS compound identities cannot be established.

PFOA was conclusively not present in any emissions. No other "legacy" PFAS compounds commonly targeted for analysis (e.g., EPA Method 537 compounds such as PFOS, PFBS, etc) were found in emissions samples.

Table 5 presents a summary of the results from the multiple blanks that were collected and integral to interpreting the impinger data. Table 6 presents the results from emission samples 1, 3, 5, 7, 9, 11, 13, and 15. Table 7 presents the results from emission samples 2, 4, 6, 8, 10, 12, 14, and 16. These data are separated to aid in the interpretation of results. These results indicate that the PFAS compounds tentatively identified were only present in sample 1. Although PFAS were also detected in samples 3, 5, 7, 9, 11, 13, and 15, this is a result of equipment train contamination (see below). PFAS compounds are conclusively not present in samples 2, 4, 6, 8, 10, 12, 14 and 16, and their absence in sample 2 indicates that the 15 PFAS tentatively identified are only present in the process emissions collected by sample 1. As a result, these 15 PFAS compounds are emitted very early in the oven heating cycle.

The fact that the 15 PFAS compounds were present in samples 3, 5, 7, 9, 11, 13, and 15 is due to contamination of the Train 2 glassware (impingers) following the collection of sample 1. The impinger samples were collected with two separate and clearly identified sets of glassware. Samples 1, 3, 5, 7, 9, 11, 13, and 15 were collected with Train 2 and samples 2, 4, 6, 8, 10, 12, 14, and 16 were collected with Train 1. Both sets of glassware were confirmed to be relatively free of contaminants prior to sampling (the proof blanks). The subsequent field blanks (blanks from glassware used for sampling) indicate a contaminant carryover in Train 2 only.

In summary, this study has characterized the emissions from the subject PTFE sintering process. Sixteen separate, 2h emissions samples were collected over the course of the ~70h heating process for TO-15 VOCs, PTFE thermal degradation products, and hydrophilic PFAS compounds. The CIMS, a real-time measurement, also characterized select PFAS compounds as a function of time over the complete heating process. The qualitative, targeted measurements from the SUMMA samples identified 39 TO-15 VOC compounds and 2 thermal degradation products (TFE and 4:2 telomer alcohol) present in process emissions. HFP and PFIB were not present in process emissions. The CIMS results identified the presence of C2, C3 and C4 PFCAs. Non-targeted analyses of the impinger samples detected and tentatively identified 15 polyfluorinated compounds present in process emissions. It was determined that these PFAS compounds were only present in sample 1, indicating that they were emitted very early in the heating process. PFOA was not identified in any of the 16 impinger process emissions samples.

Table 3. Qualitative TO-15 Results from SUMMA® Canisters – Compounds Identified with Associated Area Counts

Inst Blank Inst Blank Trip Blank Field Blank Ambient System Blank Sample 1 Sample 2 Sample 3 Sample 3 Sample 6 Target Compounds Area Counts Area Counts	Area Counts 3,131,553 4,129,583
Propylene 44,873 35,831 46,060 88,408 1,513,487 185,444 1,312,320 13,065,125 3,948,004 3,647 Propane 30,993 26,693 33,478 78,041 1,959,962 138,047 1,566,150 18,396,585 5,332,395 4,973 Dichlorodifluoromethane 1,804 1,653 3,270 4,481 76,389 7,126 72,121 84,100 91,505 82 Butane 8,010 5,777 12,088 23,353 115,427 67,537 194,930 328,668 175,836 675 Ethanol 6,545 11,997 38,102 8,879,870 45,738,075 4,774,153 103,085,831 183,226,239 143,396,317 189,383 Acetone 63,628 39,576 271,224 424,774 2,998,060 1,118,499 3,168,034 3,266,843 3,201,573 5,081	3,131,553 4,129,583
Propane 30,993 26,693 33,478 78,041 1,959,962 138,047 1,566,150 18,396,585 5,332,395 4,973 Dichlorodifluoromethane 1,804 1,653 3,270 4,481 76,389 7,126 72,121 84,100 91,505 82 Butane 8,010 5,777 12,088 23,353 115,427 67,537 194,930 328,668 175,836 675 Ethanol 6,545 11,997 38,102 8,879,870 45,738,075 4,774,153 103,085,831 183,226,239 143,396,317 189,383 Acetone 63,628 39,576 271,224 424,774 2,998,060 1,118,499 3,168,034 3,266,843 3,201,573 5,081	4,129,583
Dichlorodifluoromethane 1,804 1,653 3,270 4,481 76,389 7,126 72,121 84,100 91,505 82 Butane 8,010 5,777 12,088 23,353 115,427 67,537 194,930 328,668 175,836 675 Ethanol 6,545 11,997 38,102 8,879,870 45,738,075 4,774,153 103,085,831 183,226,239 143,396,317 189,383 Acetone 63,628 39,576 271,224 424,774 2,998,060 1,118,499 3,168,034 3,266,843 3,201,573 5,081	
Butane 8,010 5,777 12,088 23,353 115,427 67,537 194,930 328,668 175,836 675 Ethanol 6,545 11,997 38,102 8,879,870 45,738,075 4,774,153 103,085,831 183,226,239 143,396,317 189,383 Acetone 63,628 39,576 271,224 424,774 2,998,060 1,118,499 3,168,034 3,266,843 3,201,573 5,081	7 81,095
Ethanol 6,545 11,997 38,102 8,879,870 45,738,075 4,774,153 103,085,831 183,226,239 143,396,317 189,383 Acetone 63,628 39,576 271,224 424,774 2,998,060 1,118,499 3,168,034 3,266,843 3,201,573 5,081	
Acetone 63,628 39,576 271,224 424,774 2,998,060 1,118,499 3,168,034 3,266,843 3,201,573 5,081	721,002
	197,448,403
iso-Pentane 2.957 2.259 8.911 18.789 151.202 45.552 149.770 131.396 168.033 264	16 8,615,121
	337,773
Trichlorofluoromethane 2,137 1,449 4,298 6,040 98,432 9,615 98,030 102,816 106,460 117	130,411
Isopropyl Alcohol 5,278 2,940 9,085 68,775 358,845 98,808 577,579 1,839,740 1,920,271 735	1,423,077
1-Pentene 0 0 675 3,864 34,971 10,146 16,770 33,683 193,657 39	45,479
n-Pentane 0 835 3,417 16,749 176,706 35,379 198,204 129,506 240,010 348	26 310,223
Isoprene 689 581 890 2,158 18,899 3,161 8,939 10,984 18,840 21	19,574
trans-2-pentene 819 550 817 871 2,781 3,309 2,874 4,271 3,845 5	6,746
Methylene Chloride 696 486 770 1,537 14,800 3,712 12,325 23,574 20,560 27	13 33,202
2-Chloroprene 142 0 0 172 0 978 1,032 845 1,535 26	1,639
1-Hexene 0 0 0 146 1,681 1,213 976 2,306 0 55	12,388
Diisopropyl ether 0 0 0 7,439 44,439 10,008 439,360 781,769 429,295 1,137	30 1,321,155
Ethyl Acetate 0 0 0 676 5,523 1,102 51,899 98,618 50,772 140	00 162,672
n-Hexane 589 364 1,059 1,097 4,973 5,086 0 28,706 39,180 84	99,693
Methylcyclopentane 0 0 0 230 1,084 796 1,767 2,732 1,736	0 7,008
2,4-Dimethylpentane 0 258 537 1,561 3,938 3,796 23,186 41,558 23,190 88	99 115,453
Benzene 806 601 1,042 2,820 18,056 5,039 13,860 19,470 14,664 18	51 21,295
Carbon Tetrachloride 356 228 770 821 12,302 1,807 14,088 15,306 17,302 19	06 20,198
2-Methylhexane 0 0 0 13,706 14,250 11,066 297,410 601,617 301,482 1,259	75 1,624,485
3-methylhexane 0 0 0 10,243 9,942 11,621 236,848 512,155 249,903 1,097	1,388,435
Trichloroethene 389 156 363 438 4,874 1,270 3,453 3,353 5,324 6	20 7,701
Heptane 544 406 893 2,086 5,886 5,896 22,802 48,508 30,114 108	126,884
Methylcyclohexane 0 0 0 0 0 887 0 0 0 12	91 0
Toluene 0 0 645 3,193 8,436,350 10,747 5,687,089 703,712 193,278 149	50 104,550
	36 5,382
m-Xylene 417 0 471 625 13,445 2,332 13,153 7,063 15,376 7	75 6,292
o-Xylene 459 0 576 583 6,634 1,969 10,740 13,532 35,256 37	32 42,243
Chlorotoluenes 0 0 0 0 68,496 1,478 0 0 0	0 0
n-Propylbenzene 0 0 0 0 68,496 1,314 0 0 0	0 0
n-Decane 0 0 0 1,230 0 3,625 115,302 3,861,135 6,723,912 8,139	13,366,205
	00 0
Undecane 0 0 0 1,141 7,065 3,853 0 9,399 17,003 14	
Naphthalene 0 0 774 1,011 11,119 4,915 11,496 14,469 41,754 18	
	32 12,949

Table 3. Qualitative TO-15 Results from SUMMA® Canisters – Compounds Identified with Associated Area Counts (cont.)

1 abie 3. Quantati	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10	Sample 11	Sample 12	Sample 13	Sample 14	Sample 15	Sample 16
Target Compounds	Area Counts										
Propylene	3,293,739	2,466,715	3,257,166	2,998,869	2,859,171	1,955,235	1,328,651	1,419,704	2,254,872	1,500,995	2,348,850
Propane	4,332,342	2,924,116	3,847,440	3,719,995	3,616,743	2,621,187	1,589,339	1,689,416	2,752,104	1,795,543	2,941,506
Dichlorodifluoromethane	77,718	70,412	74,754	75,591	71,031	8,583	67,835	56,514	61,988	61,636	63,869
Butane	540,420	460,959	310,378	293,127	171,427	0	159,071	290,064	410,117	173,005	157,676
Ethanol	163,241,987	202,959,150	173,184,324	150,531,841	144,636,280	119,662,402	133,395,596	99,034,654	128,610,283	65,267,095	75,500,618
Acetone	6,772,819	9,240,381	12,533,772	7,920,865	5,981,315	4,343,070	2,574,875	3,298,849	4,227,614	2,339,377	2,836,831
iso-Pentane	351,023	416,807	506,403	354,067	296,114	194,825	135,679	210,934	326,239	150,768	194,981
Trichlorofluoromethane	131,941	119,770	102,476	92,191	87,887	79,071	80,800	81,978	77,185	78,057	74,921
Isopropyl Alcohol	1,230,852	2,713,686	1,876,961	3,937,491	1,747,191	2,611,589	366,014	1,049,776	2,254,911	763,009	2,236,040
1-Pentene	36,664	65,170	82,320	58,735	56,330	37,192	31,460	26,601	41,590	74,453	24,780
n-Pentane	390,767	404,945	410,551	343,644	393,694	196,708	126,878	271,967	401,236	237,060	288,258
Isoprene	21,216	20,412	24,566	32,716	26,660	17,177	8,967	7,546	31,339	7,873	24,313
trans-2-pentene	8,582	12,004	18,582	21,579	10,747	6,806	4,501	5,366	14,293	6,078	9,043
Methylene Chloride	22,541	31,883	21,870	14,897	18,045	13,381	85,888	14,646	14,491	13,055	14,596
2-Chloroprene	1,861	2,360	3,525	7,339	3,072	1,455	1,277	0	1,911	724	1,393
1-Hexene	3,634	17,316	6,325	5,257	3,514	2,011	1,584	1,673	2,724	2,376	3,066
Diisopropyl ether	727,625	1,510,143	780,677	363,624	335,601	174,629	198,999	220,114	292,753	68,310	72,930
Ethyl Acetate	85,714	187,967	96,859	42,597	38,619	20,030	23,268	24,547	35,228	8,996	9,923
n-Hexane	87,701	65,225	35,564	21,496	48,283	0	0	0	16,579	0	0
Methylcyclopentane	4,925	8,109	5,297	4,111	2,013	1,755	1,406	2,400	3,117	1,231	1,805
2,4-Dimethylpentane	64,023	136,070	43,403	15,177	9,602	5,735	4,067	20,175	16,058	2,573	6,427
Benzene	35,951	74,197	103,639	72,700	63,589	24,274	27,831	25,457	47,123	24,743	24,439
Carbon Tetrachloride	21,317	19,354	15,533	11,976	13,026	10,976	13,005	11,094	10,412	11,121	10,843
2-Methylhexane	890,756	1,955,178	739,884	388,540	262,513	138,042	44,548	219,631	170,556	9,642	20,407
3-methylhexane	748,652	1,628,757	551,099	157,119	120,588	57,027	43,107	182,025	150,199	9,396	18,929
Trichloroethene	9,868	8,463	6,978	9,758	6,809	4,523	2,898	3,132	12,944	4,139	6,685
Heptane	71,842	150,722	57,059	22,462	18,042	9,733	8,884	18,470	21,568	4,199	10,258
Methylcyclohexane	8,132	17,360	8,451	4,528	0	0	0	0	7,470	0	0
Toluene	162,039	189,926	114,371	555,892	1,485,164	140,790	2,211,207	241,099	3,058,414	748,162	11,368,465
Ethylbenzene	5,892	8,170	6,804	19,117	7,848	3,589	8,062	3,677	9,532	4,450	16,659
m-Xylene	9,139	11,464	12,426	30,044	10,838	4,849	9,859	5,233	12,939	6,178	30,642
o-Xylene	44,946	41,084	22,167	21,931	9,920	4,418	6,263	3,808	8,524	4,779	20,181
Chlorotoluenes	0	0	5,263	0	0	0	0	0	0	13,549	33,880
n-Propylbenzene	0	0	0	0	0	0	0	0	0	13,549	33,880
n-Decane	15,727,491	20,043,891	21,327,496	8,838,854	1,856,678	284,633	34,568	30,189	17,408	7,895	12,092
1,2,3-Trimethylbenzene	27,092	26,575	31,031	19,499	8,204	4,698	2,855	1,951	4,640	2,462	7,808
Undecane	37,838	35,496	69,842	31,312	9,845	5,958	5,199	4,139	0	3,594	5,588
Naphthalene	29,167	12,809	16,539	50,907	28,084	10,420	5,414	6,114	6,841	7,298	6,943
Dodecane	55,747	82,591	100,488	62,662	89,564	16,104	6,500	7,897	7,411	7,781	7,768

Table 4. Targeted TFE and Telomer Alcohols – with Associated Area Counts

		4:2 fluorotelomer
Target	TFE	alcohol
Compounds	Area Counts	Area Counts
System Blank	1,363,994	0
Field Blank	209,616	1,601
Trip Blank	905,074	0
Ambient	767,244	0
Sample 1	3,263,269	8,816
Sample 2	1,635,088	24,170
Sample 3	1,112,791	14,648
Sample 4	1,510,977	62,473
Sample 5	791,857	70,496
Sample 6	1,360,932	38,139
Sample 7	1,504,011	106,613
Sample 8	2,361,816	20,520
Sample 9	1,220,128	0
Sample 10	2,610,103	0
Sample 11	2,621,143	0
Sample 12	15,207,595	0
Sample 13	13,819,373	9,319
Sample 14	13,280,328	10,148
Sample 15	8,559,907	0
Sample 16	13,565,851	0



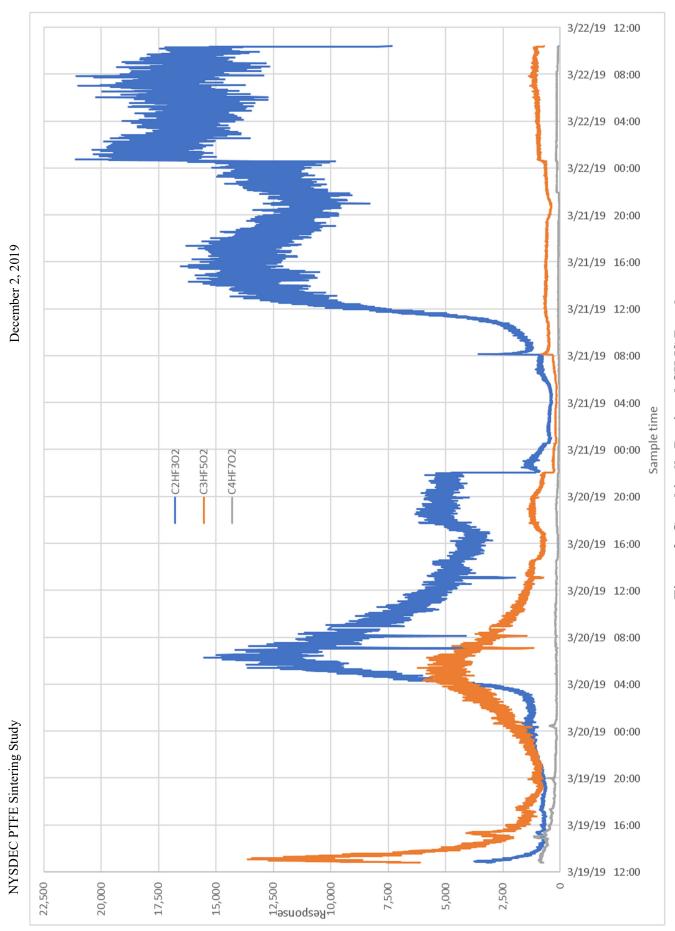


Figure 2. Graphically Depicted CIMS Results

Table 5. Non-Targeted PFAS Impinger Results – Compounds Identified with Associated Area Counts

	Reagent MeOH	Reagent Water	Proof Blank	Proof Blank	Field Blank	Field Blank 1	Field Blank 2	Nitrogen System Blank
			Train 1	Train 2	Train 1	Train 2	Train 2	Train 1
Formula	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts
C11H7F15O4	434	303	22,737	20,121	55,535	396,422	230,067	6,261
C13H9F17O4	471	495	823	811	1,116	219,924	204,015	2,420
C18H12F22O4	921	584	600	530	568	328,579	263,548	1,216
C15H11F19O4	252	159	247	226	339	111,135	145,063	579
C16H10F20O4	3,448	1,540	947	2,003	714	290,494	600,512	1,392
C9H5F13O4	572	4,244	615	4,240	1,018	364,699	93,143	2,545
C14H8F18O4	696	3,430	491	397	479	150,515	269,849	1,344
C12H13ClF12O7	479	452	997	805	2,235	62,007	69,606	2,349
C12H7F17O4	2,142	614	868	995	1,097	57,837	48,951	866
C12H4F20O7	313	258	2,788	459	1,907	318,115	744,483	5,453
C8H9F9N4O9S2	4,500	2,671	873	323	402	128,249	268,747	1,383
C11H5F13O4	324	402	898	1,074	868	26,207	36,585	1,341
C10H2F16O4	261	798	986	637	654	73,320	124,700	1,284
C14H7F17O4	472	382	501	412	611	72,474	122,861	1,487
C16H9F26O4P	12,442	874	277	470	616	191	1,378	215

Table 6. Train 2 Sample Results – Compounds Identified with Associated Area Counts

			SAMPLE 1	SAMPLE 3	SAMPLE 5	SAMPLE 7	Field Blank 2	SAMPLE 9	SAMPLE 11	SAMPLE 13	SAMPLE 15
Tentative Identification	Neutral Mass	Formula	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts
Polyfluorinated Methyl Ester	488.01113	C11H7F15O4	6,657,458	2,897,711	3,040,281	935,850	230,067	322,222	326,127	775,940	425,894
Polyfluorinated Methyl Ester	552.02388	C13H9F17O4	6,102,196	1,699,905	1,661,338	614,575	204,015	152,918	238,232	447,115	183,283
Polyfluorinated Di-Acid	710.03979	C18H12F22O4	3,663,079	722,041	1,815,089	1,054,571	263,548	139,966	408,273	842,347	11,036
Polyfluorinated Methyl Ester	616.03641	C15H11F19O4	3,176,239	646,156	876,144	311,218	145,063	65,342	126,284	215,991	55,812
Polyfluorinated Di-Acid	646.02705	C16H10F20O4	2,957,655	1,636,440	1,424,529	877,979	600,512	134,151	408,419	1,242,858	63,947
Polyfluorinated Methyl Ester	423.99841	C9H5F13O4	1,606,702	2,010,592	1,282,317	569,953	93,143	264,123	191,428	432,503	379,980
Polyfluorinated Di-Acid	582.01444	C14H8F18O4	1,977,589	990,485	567,782	405,991	269,849	94,663	209,782	497,360	48,629
Polyfluorinated Methyl Ester	532.01765	C12H13ClF12O7	1,873,588	277,380	497,234	207,658	69,606	41,856	83,972	127,151	58,011
Polyfluorinated Di-Acid	538.00816	C12H7F17O4	1,554,081	412,882	336,178	153,453	48,951	39,058	59,029	115,767	55,580
Polyfluorinated Methyl Ester	639.96115	C12H4F20O7	1,449,245	413,398	914,355	590,579	744,483	132,756	373,496	680,681	8,784
Polyfluorinated Methyl Ester	539.96721	C8H9F9N4O9S2	1,348,053	316,192	486,826	288,797	268,747	68,459	196,222	481,852	18,974
Polyfluorinated Methyl Ester	447.99855	C11H5F13O4	1,231,393	75,124	116,148	71,855	36,585	15,300	49,264	120,815	21,962
Polyfluorinated Di-Acid	489.97023	C10H2F16O4	1,178,157	263,256	307,750	171,416	124,700	35,701	103,625	292,020	16,273
(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-											
Heptadecafluorodecyl) hydrogen maleate	562.00837	C14H7F17O4	1,091,923	613,026	326,441	214,627	122,861	49,923	118,957	266,536	25,239
6:2 Fluorotelomer phosphate diester	789.98347	C16H9F26O4P	215	6,295	1,037	2,714	1,378	3,279	1,396	1,111	18,485

Table 7. Train 1 Sample Results – Compounds Identified with Associated Area Counts

		_	SAMPLE 2	SAMPLE 4	SAMPLE 6	SAMPLE 8	Field Blank	SAMPLE 10	SAMPLE 12	SAMPLE 14	SAMPLE 16
Tentative ID Name	Neutral Mass	Formula	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts	Area Counts
Polyfluorinated Methyl Ester	488.01113	C11H7F15O4	309	742	327	639	55,535	511	520	549	536
Polyfluorinated Methyl Ester	552.02388	C13H9F17O4	348	328	391	500	1,116	535	1,663	571	1,060
Polyfluorinated Di-Acid	710.03979	C18H12F22O4	658	440	558	511	568	1,034	423	441	527
Polyfluorinated Methyl Ester	616.03641	C15H11F19O4	207	209	176	206	339	332	275	241	426
Polyfluorinated Di-Acid	646.02705	C16H10F20O4	2,811	328	1,247	1,000	714	1,047	584	1,661	1,201
Polyfluorinated Methyl Ester	423.99841	C9H5F13O4	1,152	3,007	6,136	4,523	1,018	832	1,097	3,564	840
Polyfluorinated Di-Acid	582.01444	C14H8F18O4	738	207	367	3,753	479	672	1,672	599	602
Polyfluorinated Methyl Ester	532.01765	C12H13ClF12O7	579	262	387	461	2,235	500	1,603	469	871
Polyfluorinated Di-Acid	538.00816	C12H7F17O4	928	463	585	413	1,097	538	356	289	570
Polyfluorinated Methyl Ester	639.96115	C12H4F20O7	624	149	221	263	1,907	295	916	160	334
Polyfluorinated Methyl Ester	539.96721	C8H9F9N4O9S2	346	219	293	7,808	402	436	393	7,283	573
Polyfluorinated Methyl Ester	447.99855	C11H5F13O4	923	327	500	561	868	710	606	574	682
Polyfluorinated Di-Acid	489.97023	C10H2F16O4	795	296	409	489	654	673	522	617	608
(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-											
Heptadecafluorodecyl) hydrogen maleate	562.00837	C14H7F17O4	437	298	379	738	611	685	613	486	614
6:2 Fluorotelomer phosphate diester	789.98347	C16H9F26O4P	562	947	1,598	2,466	616	3,520	632	2,369	243