EXHIBIT D.

Draft Final Report

Dispersion Processor Material Balance Project

January 2005

Prepared by

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On Fluoropolymer
Safety

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Dispersion Processor Materials Balance
Final Report

I. Executive Summary

On March 14, 2003, the Fluoropolymers Manufacturers Group (FMG) of The Society of the Plastics Industry, Inc. (SPI) wrote to the U.S. Environmental Protection Agency (EPA) providing information about the efforts the members of the FMG to develop information on the uses of fluoropolymers made with perfluorinated surfactants, a class of chemicals that had come to EPA's attention in 2000. EPA's interest arose because of data provided to the agency on one such substance, perfluorooctanoic acid (PFOA), and the fact that minute amounts of PFOA had been detected in human blood in the general population.

One of the commitments included in that letter was to conduct a study of the use of aqueous fluoropolymer dispersions (AFD) at the next stage of the distribution chain, the processors who use or apply AFD in making products. The FMG decided to study this group of processors because AFD were known to contain small amounts of a PFOA salt known as ammonium perfluorooctanoate (APFO). Given the presence of APFO in AFD, the FMG recognized it was important to understand how the AFD are processed and whether such processing could be a significant exposure pathway to the general population. This Report contains the results of the Study.

- The Report is organized in eight sections and seven appendices: Section I is this Executive Summary.

- Section II of the Report provides background information, including the objective of the Study, *i.e.,* to develop a representative material balance for the fate of APFO contained in AFD.

- Section III of the Report describes the Study in detail, including (1) the roles of the members of the Study Team, (2) an overview of the Study, (3) a description of AFD processing and (4) the characteristics and demographics of the Study participants and how they were selected. Study participation was entirely voluntary. The Study participants represent approximately 50% by volume of the fluoropolymer dispersion industry. The industry segments in the Study represent approximately 75% of the annual fluoropolymer dispersion volume in the United States. Thus, based on the level of participation and an assessment of the characteristics of participants compared to the characteristics of the industry, the Study is representative of the industry as a whole. The resulting data can be used to assess the potential contribution of AFD processing to pathways of human and environmental exposure to PFOA.

- Section IV of the Report describes the confidentiality requirements of the Study. To encourage participation, the Study was designed to ensure that all sensitive business information is kept confidential to the extent permitted by law. The identity and trade secrets of participants were only disclosed to the members of the Study team to the extent necessary to achieve the objectives of the Study. These details have not been provided to any person outside the individuals directly involved in conducting the Study. Recognizing that the credibility of the Study depends in part on a degree of transparency as to the methods used and the results and data supporting the conclusions, the Study Protocol and Quality Assurance Project Plan (QAPP) were provided to the participants in the public EPA process on PFOA. In addition, EPA will be provided with Confidential Business Information (CBI) necessary to review adequately the supporting data, analytical results, and calculations, and the conclusions of the Study. The individual sampling and analytical data supporting documentation will be provided to EPA in a form that does not identify individual sites or companies. All such information is provided to EPA as CBI not subject to Freedom of Information Act disclosure under TSCA Section 14 and the regulations at 40 CFR.

- Section V of the Report describes the data collection under the Study, including the survey and sampling plan, and Section VI of the Report describes the extensive quality assurance and quality control procedures in the Study. More detail on these two subjects is included in the Study Protocol and Quality Assurance Project Plan (QAPP), which are attached in Appendices I and II. Extensive quality control procedures were established, including spiking of samples with dual ^{13}C labeled PFOA to measure recovery efficiencies for the analytical work performed. A copy of the survey form is included in Appendix IV. Participants provided some of the data used in the Study on the survey form, while other data were collected during on-site sampling at processors' facilities.

- Section VII of the Report describes the industry processes incorporated in the Study, including, "Glass Cloth Processing," "Formulating Coating Products," "Metal Coatings," and "Additives."

- Section VIII of the Report provides the results and conclusions of the Study. The Study results are reported as PFOA Partition Factors (PPFs) for the individual process segments observed in the studied processes. In addition, estimates are provided of the proportion of APFO contained in AFD that might be (1) present in different environmental media, (2) destroyed in the process, or (3) used in processes that were not represented in the Study. These amounts are also expressed as a fraction of the total amount of APFO used in making fluoropolymers by the FMG members.

The data collected in the Study support the conclusion that very small amounts of APFO can be found in air, wastewater and solid waste streams. These results and data from publicly available sources suggest that most of the APFO used of AFD is destroyed in processes where products are sintered; *i.e.*, where temperatures are sufficiently high for a sufficient length of time to destroy the APFO present. Based on the results of the sampling and analysis, 62% of the APFO from AFD is destroyed and approximately 25% ends up in air, water, and solid waste streams. Most of the remaining 12%, which represents less than 2% of total APFO, is used in processes where sufficiently high temperatures are not reached. Whether any of the APFO in AFD is present in products will be the subject of the Articles of Commerce Study.

II. Introduction

On March 14, 2003, the Fluoropolymers Manufacturers Group (FMG) of The Society of the Plastics Industry, Inc. (SPI) wrote to the U.S. Environmental Protection Agency (EPA) providing information about the efforts the members of the FMG to develop information on the uses of fluoropolymers made with perfluorinated surfactants, a class of chemicals that had come to EPA's attention in 2000. EPA's interest arose because of data provided to the agency on one such substance, perfluorooctanoic acid (PFOA), and the fact that minute amounts of PFOA had been detected in human blood in the general population.

One of the commitments included in that letter was to conduct a study of the use of aqueous fluoropolymer dispersions (AFD) at the next stage of the distribution chain, the processors who use or apply AFD in making products. The FMG decided to study this group of processors because AFD were known to contain small amounts of a PFOA salt known as ammonium perfluorooctanoate (APFO). Given the presence of APFO in AFD, the FMG recognized it was important to understand how the AFD are processed and whether such processing could be a significant exposure pathway to the general population. This Report contains the results of the Study.

The manufacturers of fluoropolymers use a chemical called ammonium perfluorooctanoate (APFO, also known as C8) in the manufacture of some fluoropolymers and fluoroelastomers in the United States. Fluoropolymers are plastic products while fluoroelastomers are rubber-like products, both of which possess highly desirable and unique properties that make the articles of commerce created from them useful. APFO is used to suspend and emulsify some fluoropolymers during manufacture and small amounts can remain in the aqueous fluoropolymer mixtures called dispersions. Certain fluoropolymers made with APFO are used in high-performance applications in critical industries such as defense, aerospace, semiconductors, telecommunications, and pollution control. Throughout the remainder of this document, the term "APFO" will be used to represent both PFOA and APFO unless the context requires that we specify the acid form.

In 2001, the FMG provided EPA with information on what happens to APFO used in the members' manufacturing processes. The fluoropolymer manufacturers material balance Study showed that approximately 15% of the APFO used to make fluoropolymers world-wide remained in products sold to customers in the form of AFD.[1] The APFO content of AFD was reported to be on the order of less than one-half percent by weight and is only present because it is left over from the fluoropolymer manufacturing process. In a letter to EPA Assistant Administrator Stephen L. Johnson, dated March, 2003,[2] the FMG member companies announced their intention to conduct a study called The Dispersion Processors Material Balance (DPMB) Study, which would examine the processes and characterize the passage of APFO from AFD through customers' facilities. Specifically, these fluoropolymer manufacturers agreed to:

> Engage a third-party consultant to develop a representative material balance for the fate of APFO contained in these dispersions. Similar to the information provided to EPA on fluoropolymer manufacturing, address in the representative material balance how the dispersion is used at the customer site and potential emissions of APFO to the environment. . . .

This Report contains the results of that Study.

A. <u>The Objective of the Study</u>

The objective of the Study was to understand how APFO contained in AFD that are used in processing plants might find its way into the environment. More specifically, data were to be collected describing the potential contribution of dispersion processing to possible environmental pathways of exposure to APFO from air, water, and solid waste media. The method chosen in the protocol was to develop PFOA Partition Factors (PPFs) based on the process characteristics specific to these dispersion-processing industry segments.

The Study used a survey and sampling method to complete a material balance for APFO during processing of these dispersions. The FMG members identified four categories of processes prior to the beginning of the Study. The intent was to select participant facilities for sampling that would allow collection of representative data in each of the categories. The data would then be used to characterize the various dispersion-processing industry segments by (1) the amounts of APFO used and (2) the amounts of APFO potentially entering environmental media from dispersion-process operations.

The processes selected for Study are representative in that they exhibit characteristics which allow a comparison of like operations to predict what happens to the APFO when the AFD are applied to various substrates to make finished and semi-finished goods. Industry segments were identified and the Study was designed to collect data from each of these processes.

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[1] The original estimate given to EPA for the fraction of APFO in AFD sold in the U.S. was based on year 2000 data. The percentages and amounts of APFO in AFD reported by manufacturers and processors included in this report are based on 2003 data. Details of the data and calculations are confidential due to the sensitive commercial nature of amounts sold by manufacturer to individual processors; the details are described in greater detail to EPA in an attachment claimed as Confidential Business Information under the Toxic Substances Control Act (TSCA).

[2] OPPT-2003-0012-0012, Letter of Intent to assist EPA in assessment of PFOA and its salts to S.L. Johnson, EPA from D.K. Duncan, SPI, March 14, 2003.

A small group of industry experts identified these processes by performing an informed analysis based on specialized knowledge of the industry and its practices, the volumes of dispersions processed, and the specific dispersion processing methods that are used. It was not an objective of the Study to collect data using a statistically based model or to produce a statistically representative compilation of data from the universe of AFD processors. The data collected were to be used to calculate the PPFs that could be used to estimate the material balance for the dispersion processing industry as a whole. Additionally, it was hoped that the PPFs could be used to predict where APFO would be found in unsampled facilities in the same category of the AFD processing industry.

The residue of APFO that may remain in finished products was not measured directly in this Study. More detailed information on the presence of APFO in articles of commerce is being provided by the FMG in another voluntary Study, and a further Study of aged articles of commerce is being developed as part of the Enforceable Consent Agreement (ECA) process. However, information available at the time of the Study and developed in the Study allowed estimates of the relative proportion of APFO that is destroyed in the processes. These estimates are based on (1) information on residues of APFO in processed fluoropolymers that is available in the open literature or in publicly available documents, (2) thermal profiles of articles made with dispersions in the Study, (3) extrapolation from analysis of certain waste materials in the Study, and (4) literature regarding the kinetics of thermal destruction of APFO.[3]

III. Description of the Study

The Study Protocol and the QAPP were provided to EPA and participants in the public ECA process prior to the Study. No comments were received from any public participants. EPA provided comments that were taken into account in preparing the final protocol. Copies of these documents and the survey form are found in Appendices I - IV.

A. The Study Team

The FMG retained Keller and Heckman LLP (K&H) of Washington, D.C. to manage the Study on behalf of the FMG members. The specific tasks and roles assigned to the organizations performing the Study, as well as the qualifications of the Study team, are described in more detail in the Study Protocol, dated December 2003, and attached as Attachment 1. Below is a brief description of the team and their responsibilities.

K&H was responsible for (1) overall coordination of the Study at processor facilities in the United States, (2) preparation of the Study survey form, (3) arranging for the processors to participate, (4) preparation and execution of confidentiality agreements between the individual Study participants and the Study team, and (5) overseeing preparation of the final Report.

K&H retained Barr Engineering Company (Barr), a nationally known environmental engineering firm, as the primary technical consultant. Barr was responsible for (1) reviewing the

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[3] *Gas-Phase NMR Technique for Studying the Thermolysis of Materials: Thermal Decomposition of Ammonium Perfluorooctanoate*, Krusic, P.J. and Roe, D.C., Anal. Chem., 76:3800, 1 July 2004.

survey data for data gaps and adequacy for the Study's purpose, (2) developing standard protocols for collecting samples, (3) preparing the Study Protocol and Quality Assurance Project Plan (QAPP), (4) preparing facility sampling plans based on a review of the facility surveys and telephone interviews with survey participants, (5) recommending participants for sampling, (6) performing the field site visits pursuant to the Study protocol, (7) calculating the PPFs from the data, (8) preparing the technical analysis and process descriptions for the individual field Study participant reports, (9) preparing the final Report, (10) providing quality assurance in the field and in reviewing the analytical data for the final Report (11) providing a Quality Assurance/Quality Control (QA/QC) assessment of sample recovery efficiencies and the reliability of the sample results. and (12) providing overall technical support and chemical engineering expertise.

K&H retained KHA Consulting LLC (KHA) to provide industry-specific expertise and knowledge. KHA was responsible for (1) assisting in the Study design, (2) identifying and classifying the processes used in the industry, (3) preparing typical descriptions of the processes encountered, (4) assisting Barr in designing the sampling plan, (5) participating in the field site visits to oversee protocol compliance and to assure that the sampling plan gathered relevant and representative data, and (6) reviewing the final Report. KHA also prepared the process schematics contained in the final Report with the assistance of Barr.

K&H retained Exygen Research (Exygen) as the Study analytical laboratory. Exygen's role was to (1) perform sample analyses, (2) identify and provide protocols for sampling and analytical methods (3) develop and validate as needed sampling and analytical methods, (4) provide quality assurance in the laboratory, and (5) assist in preparation and review of the final Report.

B. Study Overview

Prior to sending the survey to each processor facility, K&H contacted each facility to inform it of the purpose and content of the Study, secure cooperation, verify the appropriate contact person(s), determine the types of processes for sampling planning purposes, preview the survey content, and negotiate the details of any confidentiality agreement requested by the processor.

Barr conducted the sampling program at processor facilities and analyzed the data to produce the PPFs. Barr evaluated the facilities by survey review and telephone interview, with the assistance of KHA. From the telephone interviews, Barr selected sites for pre-sampling site visits, subject to review by K&H, and scheduled pre-sampling site visits. Barr then prepared sampling plans, which were reviewed by the Study team, to define the types and locations of samples to be collected. Once the processes were selected and sampling plans completed, a sampling team from Barr visited each site to complete the sampling and follow up on data gaps in the survey. The sampling and analysis protocols are found in Attachment IV.

Barr collected samples of water, air, and solid wastes from each site according to the plan and used that information to calculate PPFs for specific processes that could be used to estimate the amounts of APFO (1) partitioned into various waste media from the AFD processing industry or (2) destroyed or remaining in the products. Barr shipped the samples collected to Exygen for analysis, and Exygen forwarded the analytical results to Barr for data review, reduction and interpretation.

Following receipt of the laboratory results, Barr generated a process-specific report for each sampled process and forwarded those reports to K&H. K&H sent the Processor Material Balance Reports to each processor for review and asked each processor to identify CBI and to review the process description for accuracy.

The amount of APFO contained in AFD used at each facility during sampling was obtained from the fluoropolymer manufacturer for the specific product being produced. For formulated products, the formulator provided the amount of AFD added to the coating in the formulator's process, and the APFO content was calculated based on the fluoropolymer manufacturers' data. This is discussed in greater detail in section III.D.6.

Once the individual reports were completed, they were used to prepare this aggregate Report. Original data and survey forms were returned to the processors upon completion of this Report.

C. AFD Processing

A fluoropolymer consists principally of fluorine atoms covalently bonded to carbon atoms that are attached to other carbons in a chain or backbone. The presence of this fluorine imparts to fluoropolymers their special characteristics. The carbon-fluorine bond is among the strongest in nature, much stronger than the carbon-hydrogen bonds found in traditional hydrocarbon plastics and rubbers. The physically larger fluorine atoms also do a better job of "protecting" their carbon backbones than do hydrogen atoms, making fluoropolymers more resistant to chemical attack. Furthermore, once attached to carbon, fluorine atoms are largely non-reactive and inert, even in the presence of highly reactive chemicals. As a result, fluoropolymers exhibit much higher chemical and heat resistance than hydrocarbon polymers.

The basic chemical and physical properties that make fluoropolymers attractive in diverse applications include the following: chemical resistance, thermal stability, UV and radiation resistance, cryogenic properties, low coefficient of friction, low surface energy, low dielectric constant, high volume and surface resistivity, and flame resistance. Applications for fluoropolymers take advantage of one or more of the properties that set them apart from other materials, particularly other plastics.

Fluoropolymers, including dispersions, require unique processing conditions, which usually includes a final heating process. That process, as described below, dictates the temperature and time range within which these polymers must be heated. Processing below the required range results in an article that does not have all of the important performance properties, and processing above the required range can degrade the fluoropolymer's properties. Fluoropolymer manufacturers and formulators advise their customers to control oven and part temperatures carefully.

Polytetrafluoroethylene (PTFE), fluorinated ethylene propylene (FEP), and other polymers that are sold as AFD are used to coat or impregnate various substrates by the processes discussed in detail in Section VII. Processes using AFD generally consist of the following steps:

coating or impregnation of the substrate, heating of the coated substrate to dry the article, and then further heating to *sinter* or melt the fluoropolymer to develop final properties. Typically these processes produce articles that require assembly into final industrial or consumer products.[4]

The sintering of a fluoropolymer, sometimes called "coalescence,"[5] is similar to that performed in metallurgy. Heating at temperatures in excess of the melting point of the fluoropolymer causes the polymer particles to melt and fuse together, eliminating voids that can result in weak points in the polymer structure. The time and temperature profile of the sintering cycle affect the final properties of the product.

Sintering properly to achieve final characteristic polymer properties requires that the temperature of the polymer exceed its melting point. The melting points of some of the fluoropolymers used in dispersions are shown in Table 1.

Proper sintering is not dependent only on temperature, it is also dependent on the size and thickness of the fluoropolymer object being processed and the time taken to run the sintering process. For example, PTFE must be heated to a minimum of 342 deg C (648 deg F).[7] Very large, thick pieces of PTFE are often heated to 360 - 380 deg C (680 - 716 deg F) for long periods of time, 12 or more hours, then slowly cooled so that cycle times can be 24 hours or more. The slow heating is necessary to assure that the polymer is melted through and through, without exposing the exterior layers to temperatures that would cause breakdown of the polymer.

Table 1
Melting Point of Various Fluoropolymers
Dispersions and Typical Processing Temperatures[6]

Polymer	Melting Point (deg C)	Typical Processing Temperature (deg C)
PTFE	342	380
FEP	245 - 280	360
PFA	300	380

In contrast, coatings and films are much lower in mass, hence, curing times are much shorter, and higher temperatures in the range of 420 - 450 deg C (788 - 842 deg F) for 5 -15 minutes, can be tolerated. Regardless of the sintering cycle, PTFE temperatures in excess of 500

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[4] Useful texts on the properties and processing of fluoropolymers are: Ebnesajjad, Sina. Fluoroplastics: Volume 1. Non-Melt Processible Fluoroplastics. William Andrew Publishing/Plastics Design Library (2000); Scheirs, John, Ed.. Modern Fluoropolymers. Wiley, Chichester NY(1997); Drobny, Jiri George. Technology of Fluoropolymers. CRC Press, Boca Raton FL (2001). The sintering discussion is from Fluoroplastics.

[5] *Fluoroplastics: Volume 1. Non-Melt Processible Fluoroplastics*, Ebnesajjad, Sina, William Andrew Publishing/Plastics Design Library (2000).

[6] *Guide to the Safe Handling of Fluoropolymers Resins*, The Society of the Plastics Industry, Inc. (SPI), 3rd Edition, 1998 (Safe Handling Guide), Appendix B.

[7] PTFE is unique in that it has two melting points, 327 deg C and 342 deg C (623 deg F and 648 deg F). The higher melting point occurs the first time PTFE is melted; changes in the polymer crystalline structure lower the melting point on subsequent heating. Because of the way in which aqueous dispersions are made, all PTFE in these dispersions is melted in sintering for the first time and melts at the higher temperature. See *Guide to the Safe Handling of Fluoropolymers Resins*, Chapter 3, p.6.

deg C (932 deg F) for an hour lead to significant thermal degradation of the polymer and ultimate failure of the product.[8] Other fluoropolymers have similar requirements, with different temperature profiles and limits unique to their chemical structure.

Achieving melting temperatures throughout the polymer may require processing temperatures above the melting point of the polymer, as measured by oven or substrate temperature, due to the poor heat transfer characteristics of fluoropolymers. Often the required temperature profile is determined by trial and error testing. Both the temperature and the time of the heating cycle must be optimized for the application and the substrate used. If, for example, the substrate is non-metallic, such as a plastic, higher temperatures or longer times will be required for complete sintering. If, on the other hand, the substrate is metal, which provides excellent thermal conductivity for heating and thermal transfer, lower processing temperatures and/or shorter processing times will often result in complete sintering.

This high temperature processing is significant because APFO breaks down into carbon dioxide and 1H-perfluoroheptane at elevated temperatures.[9] The decomposition rate is time and temperature dependent, with a reported half-life of 2.2 minutes at 234 deg C (453 deg F). The authors extrapolated the APFO half-life to be 0.06 sec at 350 deg C (662 deg F) and 0.005 sec at 400 deg C (752 deg F).[10] At 350 deg C, the APFO half-life is 0.14 sec, and the APFO is essentially all theoretically decomposed in one second. Thus, if a product made from or coated with AFD is sintered as part of its processing, and the fluoropolymer coating exceeds this temperature for any significant period of time, it is highly likely that substantially all of the APFO present in the coating will be destroyed, and none will be carried through in the processed article from the dispersion processor to its customers. Analysis of waste products in this Study suggests support for this conclusion, but confirming analyses of finished goods is the subject of another Study.

As discussed below in more detail, wastewater is generated, but, in most of the processes, is a relatively small total quantity. That water is often collected and sent off-site for treatment. Whether treatment removes APFO from the water was not an objective of the Study and no processors reported any data on the efficacy of water treatment methods.

D. Selection and Characteristics of Study Participants

The companies who signed the Letter of Intent sponsored the Study. As a result, the focus of the Study was limited to products supplied by the sponsors.

1. Method of Identification and Selection

The participants in the Study represent most of the segments in the fluoropolymer dispersion processing industry. Fluoropolymer manufacturers provided confidential customer lists for AFD categorized according to the scheme described below and volume of APFO used.

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[8] Ebnesajjad, p. 541.

[9] Krusic, P.J. and Roe, D.C., p. 3800.

[10] Ibid, p.3803.

The Study participants were selected by examining the consumption of AFD of each processor, and by considering several relevant factors, including (1) size of operation, (2) frequency of fluoropolymer use, (3) volume used,(4) nature and temperature characteristics of the operation, (5) geographic location, and (6) application methods. A goal of participant selection was to include both large and small processors, and this was accomplished.

The Study protocol called for the identification and selection of processors based on the relative ranking of the processors in their particular industry segments considering the criteria listed above. It was not random or based on achieving a statistically representative sample. The number who agreed to participate was such that all could be accepted into the Study.

Potential participants received a letter inviting them into the Study, and additional significant efforts were made to encourage participation, including (1) follow-up letters from the President of SPI, the Chairman of the SPI Fluoropolymers Processors Group (FPG); and SPI's outside counsel; (2) a presentation by senior EPA staff at an industry meeting; and (3) direct contact by manufacturers with processors. Those who agreed to participate after submitting the survey cooperated fully and completely without reservation. Over 50 processors were sent invitations to participate in the survey. Survey forms were received for 15 individual processes, providing data for six of the nine cells in the original matrix Table 2, as described below.[11]

2. *Geographic Location*

EPA considered geographic location an important variable in AFD processing. As hoped, Study participants are located in each of the regions of the country where AFD processing occurs. Additionally, key participants included processors in urban, suburban and rural areas. However, the change from a site-based process selection methodology makes geographic location less important. Process-based PPFs can be applied regardless of where processors are located.

3. *Processes Studied*

Based on information provided by the fluoropolymer manufacturers, AFD uses were classified by four broad categories – "Metal Coatings," "Glass Cloth Coatings," "Additives," and "All Other" – and by the highest process operating temperatures that the AFD were likely to see. The following temperature categories were used: less than 150 deg C (302 deg F); 150 - 250 deg C (302 - 482 deg F), and greater than 250 deg C (482 deg F). This resulted in a four-by-three matrix, shown in Table 2.

Processors who use AFD in the "All Other" category included those who make ("cast") or coat film and those who impregnate fabric or yarn made of various materials with fluoropolymers. These processes are described in more detail below.

The original manufacturers' material balance prepared in 2001 was based on data for a one-year period in 1999 - 2000; each manufacturer determined the specific time period they

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[11] The number of processors who participated was less than 15, as some processors operated more than one process.

reported based on either calendar year or fiscal year data. The data in the individual cells did approximately equal *in toto* the reported amounts distributed in the U.S. by the fluoropolymer manufacturers, within the limits of the survey conducted.

K&H updated the estimates of total APFO used in each industry segment based on confidential data provided by the manufacturers on their sales of dispersions to their customers, in terms of kg of APFO in the dispersions, for calendar year 2003. There was no significant change in the total amounts reported.

Table 2
Dispersion Processor Materials Balance
Annual Consumption, kg APFO 1999 - 2000

Market Segment	<150 deg C	150 – 250 deg C	>250 deg C
Metal coating formulators, coaters	1000 – 3000	No Uses[1]	No Reported Direct Sales[2]
Glass cloth coating	No Uses	No Uses[1]	>3000
Additives	100 – 1000	100 – 1000	CBI[3]
All Other	100 – 1000	CBI[3]	100 – 1000

1 – Of the total possible 12 cells in the table above, there are no processes that use APFO-containing dispersions in three cells, making the maximum nine cells. Participants represented six of the nine cells.
2 – We assume all formulated metal coatings are consumed by this category.
3 – CBI = confidential business information. See text.

The data from the manufacturers initially showed that for three cells in the table, there were no processes that use APFO-containing dispersions. These categories were eliminated from the Study. For the nine remaining categories, the data in the table represented the amount of APFO contained in AFD sold by manufacturers into that market segment estimated at the beginning of the DPMB Study.

Data for some of the segments were obtained from only one or two manufacturers or for one or two processors. Thus, disclosure of the amounts of APFO sold into that market segment would allow the manufacturers and processors to determine relative market share, and would thereby disclose confidential, market-sensitive information. To protect the confidentiality of such information, there must be at least three manufacturers <u>and</u> three processors in the category. In light of this consideration, in those segments for which either (1) fewer than three manufacturers reported sales data, or (2) manufacturers reported data for two or fewer customers, no data can be provided in the public Report. Accordingly, the cell is marked "CBI." The amounts of APFO sold into these segments are being provided to the U.S. EPA as part of a confidential attachment to this Report under the Toxic Substances Control Act (TSCA) provisions protecting CBI.

4. *Selection of Processors Included in Study*

The Study Protocol contemplated selecting participants by assessing the consumption of AFD of each processor and by considering a number of relevant factors, so that processors representing each cell in the Table 2 matrix would be included in the Study. The criteria for selecting processes to be sampled included: (1) how representative the processes were of those used by the dispersion processor industry, (2) production volume, (3) production time and capacity, (4) variation in temperature processing, and (5) processing time. The identification of processors to be solicited in the Study was subjective, in that the AFD manufacturers provided their identities confidentially to K&H. Selection for site visits and sampling was based on the relative ranking of the processors in the segment of the industry in which they participated. The selection was neither random nor based on achieving a statistically representative sample. The intent was to include both large and small processors.

A judgmental sampling approach was chosen instead of a random sampling approach. Judgmental sampling is used when there is extensive knowledge of the processes and when the population is known to have identifiable patterns.[12] Judgmental sampling is used when the goal is to predict average or typical results. With the extensive knowledge of the types of processors in this industry, and the differences between processes, judgmental sampling was used to provide for a balanced number of process representatives from the four product categories, including "Glass Cloth Coatings," "Additives," "Metal Coatings" (formulation and application), and "All Other." A strictly random sampling approach would run the risk of failing to include one or more important categories. The stated objectives in the Protocol to Study processes that are representative of normal operations in the industry and operations that occur on a regular basis[13] are consistent with the choice of a judgmental sampling approach.

Of the fifteen individual processes included in the Study, Barr completed sampling at eight, while four of the remaining seven provided results in the surveys based on sampling by others. No sampling data were available for the remaining three processes. Data and information from two of the three remaining processes were (1) included in the survey response forms, (2) confirmed through telephone interviews and/or pre-sampling site visits, and (3) validated based on results from sampled processes. For one processor, the survey data were examined by the engineers and compared to data from similar processes, but were not used as the sole source to derive a PPF for any process.

Barr was asked to develop a method to compare the industry to the Study participants and to evaluate the representativeness of the participants with regard to their environmental profile. That assessment is described in more detail below. Barr found that the participants were not significantly different from those processors who could be publicly identified and for whom data were available on environmental parameters. Thus, the Study participants were determined to be representative of the dispersion processing industry as a whole.

All of the volunteers who participated in the survey were considered for inclusion in the sampling phase. However, four of the original volunteers were not included in the sampling

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[12] *RCRA Waste Sampling Draft Technical Guidance,* U.S. EPA, 8/02 EPA 530-D-02-002, August 2002, pp. 53-55.

[13] *Material Balance Study Protocol,* Barr Engineering, December 2003, pp. 1, 3, and 7.

phase because the process was not operating at the time of sampling or the process or appropriate dispersion was not available for use at the time of the visit. Two of the four facilities were visited by Barr to verify data provided on survey. PPFs for all processes were based on at least one set of sampling data, either from the Study or from the survey participant. Thus, PPFs were based on sampling from processes representing approximately 57% of total reported APFO used in AFD. Based on the surveys and sampling, AFD containing approximately 75% of the APFO in AFD were used by the processors who participated in the Study.

5. *Coverage of Processes in Data Matrix*

The Study protocol called for representatives of processes from each cell in the Table 2 matrix to be included in the Study and for sampling to be done at a minimum at one site in each cell. The Study actually included facilities in six of the nine cells initially identified as having processors using AFD. There were three industry segments that were not represented in the Study population: (1) "Additives," processing below 150 deg C (482 deg F); (2) "Additives" between 150 and 250 deg C (302 - 484 deg F); and (3) "All Other" between 150 and 250 deg C (302 - 484 deg F). For these segments, estimates of APFO in input AFD were possible and by comparison of publicly available descriptions of the uses and of the processes that were studied, a judgment could be made about what happens to the input APFO in these segments could be made. These are discussed in the Results section below.

In addition to ensuring coverage of the different processes in the industry, the data and review of individual processors allowed an examination of the original classification scheme to determine if the categories originally created could be consolidated or if the definitions of the categories, in particular the temperature classification, was an accurate predictor of what happened to APFO in processing. The several changes that resulted from the examination are discussed in the Results section. Of note, for one category, Additives above 250 deg C (484 deg F), the data collected from the manufacturers and information generated during the Study resulted in the reclassification of the processors originally included in that category into other categories. That left no processors in that category. Table 3 shows the updated 2003 distribution of AFD use in the format of the original table. The revised classifications account for 100% of the total APFO in AFD sold by companies who signed the LOI.

Table 3
Dispersion Processor Materials Balance
Annual Consumption, % of Total APFO in AFD, 2003

Market Segment	<150 deg C	150 – 250 deg C	>250 deg C
Metal Coating Formulators, Coaters	10-15	No Uses	No Reported Direct Sales[1]
Glass Cloth Coating	No Uses	No Uses	50-55
Additives	CBI	CBI	No Uses
All Other	CBI	CBI	CBI

[1] We found no uses of formulated metal coatings containing fluoropolymers other than processes where sintering is the final step. Therefore, all AFD used in making metal coatings is processed at high temperatures.

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6. *Amounts of APFO Contained in AFD*

At the time of the Study, AFD commercially available from the LOI signers contained a maximum of 7 kg (15.4 pounds) of APFO per 1000 kgs (2205 pounds) of dry fluoropolymer resin, or 0.7% by weight, and typically contained less than 0.2%. The lowest reported amount of APFO in AFD was 0.04%. The accuracy of the fluoropolymer manufacturers' APFO content was ±10%, (as reported by the manufacturers). The fluoropolymer manufacturers consider the exact amount of APFO contained in AFD to be CBI. Calculating a true weighted average of APFO content of AFD was not possible with the data available. However, a simple numerical average of available data, adjusted for relative market share, shows that the APFO content was 0.28%.[14] This conclusion is based on data supplied by the manufacturers who supported the Study.

No participants in the Study reported adding APFO to any of the AFD used in their processes. Given that the Study participants are representative of all known market-segments of the dispersion processing industry, it appears reasonable to conclude that both (1) use of APFO as an additive does not occur in the dispersion processing industry and (2) all APFO present in the dispersion processing industry stems from the APFO present as an unintentional residual in the fluoropolymer dispersion products used by the industry.

7. *Demographics of the Processors Included in the Study*

Facility size varied from very small operations (less than 25,000 square feet (sf)) involving a few people to large facilities (greater than 100,000 sf) employing up to several hundred people. All of the individual sites of processors in the industry fall within these demographic parameters.

IV. Confidentiality

To encourage participation, the Study was designed to ensure that all sensitive business information is kept confidential to the extent permitted by law. The identity and CBI of participants were disclosed to only the Study team and have not been provided to any person outside the individuals directly involved in conducting the Study. Recognizing that the

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[14] Data on the APFO content of dispersions used by processors in the Study were provided on a product-specific basis by the FMG members who supported the Study. Each manufacturer's "weighted average" was calculated by averaging the available information on APFO content (as a percentage of dry polymer weight in the dispersion) and using data on amounts of APFO in AFD sold to its individual customers. Multiplying the percentage of the total AFD supplied by an individual manufacturer by that manufacturer's average APFO content and summing those results provided an estimate of the "weighted average industry APFO content." We believe this leads to a more accurate estimate of the amount of APFO used in the industry when combined with the data on APFO dispersion product use reported by the processors. All of the data used to calculate this value is CBI because it contains sensitive business information directly related to market share and individual manufacturers' customer lists.

credibility of the Study depends in part on allowing EPA to review the supporting data, analytical results, and calculations, the FMG and the participants agreed to provide the individual sampling and analytical data to EPA in a form that does not identify individual sites or companies. EPA acknowledged that the need for the data and the importance of encouraging participation warranted taking a confidential approach. Toward that end, the Study team entered into confidentiality agreements with Study participants.

The survey and data collection forms were coded and the identities of the Study participants were kept apart from the data forms. Reports were prepared with detailed data broken down only to the extent necessary to provide accurate information. Participants did not include any identifying information on the data collection forms and were asked to clearly mark any information that they deemed CBI. Reports of the results of individual participant analyses were coded and provided only to the participants for review of accuracy and identification of proprietary data, and were retained only until the completion of this final Report. All survey forms, draft reports, final reports, and other individually identifiable data were returned to the participants at the end of the Study, and the participant identity key was destroyed.

V. Data Collection

The collection of data for the Study took two forms, a survey and on-site sampling of processes. Each processor initially received a survey form asking for information on the use of APFO, details of processing, polymers used, and emissions/effluents from their facilities. Prior to sending the survey, K&H contacted each processor facility to inform it of the purpose and content, secure cooperation, verify the contact person(s), determine the types of processes for sampling planning purposes, preview the survey content, and schedule a time to conduct a telephone interview. Results from the survey were used to structure the on-site sampling program that was conducted at individual facilities.

A. Survey

The survey form, shown in Attachment III, included three primary parts: (1) specific product category and total quantity of APFO materials used by the facility, total quantity of APFO in the product sold, and total quantities of APFO destroyed and released to air, water, and solid waste; (2) site-wide handling of discharges to the air, water and solid waste; and (3) specific processes and process conditions. The survey inquired whether a facility uses materials potentially containing APFO other than AFD, or whether a facility intentionally added APFO. None of the facilities reported adding additional APFO to their processes. None of the processes sampled had materials potentially containing APFO other than AFD.

After the surveys were completed by each processor, a telephone interview was conducted with each processor to review the survey, understand the safety procedures applicable to sampling, discuss which processes would be sampled, and identify the sample collection locations. The purpose of the survey and telephone interview was to collect as much readily available information as possible from the processors and identify missing data that would be necessary to complete the Study. The survey and subsequent conversations formed the basis for selecting individual processes for sampling that were either the most representative on an annual basis or that helped to define the reasonable maximum actual emission scenarios.

Barr and KHA analyzed the survey data to determine whether quality control and assurance objectives were met, identify data gaps and sampling needs, and select representative or maximum actual emissions process conditions if sampling was required.

B. Sampling Plan

The sampling plans were devised to support the development of PPFs. PPFs enable the extrapolation of sampling data obtained at a limited number of individual processor locations to an industry-wide process-based material balance. PPFs identify process-specific parameters that describe emissions to the environment. The industry-wide, process-specific material balance supports the Study objective of accounting for APFO from AFD.

1. Determining Sampling Sites

Sampling sites were selected for their specific utility in the identification of parameters that determine where APFO may finally go in any given process. More specific details as to how the sampling locations within an individual process were selected may be found in Section B of the QAPP and in the Barr Standard Operating Procedures (Appendix B of the QAPP).

2. Process Specific Sampling

The process specific sampling protocols were based on the assumption that APFO released from the product during processing and not destroyed is carried out of each individual process in the process air, stack gas, water, or solid waste. In processes where the products are sintered, the data suggest that minimal APFO remains in the finished sintered product. These data include the half-life/temperature profile of APFO and/or the amount of APFO measured in heat-treated product-trim waste. In unsintered products, APFO may remain in the product after processing. Sampling protocols for each of the environmental media are summarized below and described more fully in Attachment IV.

3. Air Sampling

An apparatus consisting of a filter and solid sorbent material contained within a narrow gauge tube was employed to collect ambient air samples in and around the process location(s) and air samples from small diameter, low (ambient) temperature process ducts. Samples were collected using an air sampling pump drawing air through the sampling tube at a constant (non-isokinetic) rate. The sampling tube filtered the air stream and the solid sorbent material preferentially retained PFOA for subsequent analysis by a High Performance Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) analytical procedure described in Attachment IV. Detailed sampling procedures may be found in Attachment IV.

High temperature (above ambient) stack gas samples, as well as samples from larger diameter gas streams flowing in ducts, were collected isokinetically according to the Barr Standard Operating Procedures (SOPs) derived from EPA stack gas sampling methods. A measurement site was selected at a location at which laminar flow is anticipated. The samples were drawn isokinetically at multiple traverse points into the sampling train. The sampling train consisted of a heated probe, several chilled glass impingers,[15] and a glass fiber filter that was maintained at room temperature to collect both solid and gas phase chemicals present in the stack air stream.

APFO mass was determined by an LC/MS/MS analytical method described in the Study Protocol (Attachment I) and included contributions from APFO condensing in the sampling train, scrubbed in the impingers, or chemically reacting with the sample train impinger solutions and that filtered from the gas stream.

Other gas parameters were measured according to the methods in Attachment IV that contributed to the calculation of the APFO mass rate in the duct(s) or stack(s), including volumetric airflow rate, oxygen, nitrogen, and carbon dioxide concentration, and moisture content.

4. *Solid Waste Sampling*

Specific sampling locations are described more fully in Section 7 but, in general, samples are taken from tanks and sumps servicing process outflows, off-specification products, filter materials, and processing equipment. APFO concentration was then determined by the LC/MS/MS analytical method. Detailed sampling procedures are found in Attachment IV.

5. *Water Sampling*

Specific sampling locations are described more fully in Section 7 but, in general, samples were taken from collection areas such as tanks. APFO concentration was then determined by the LC/MS/MS analytical method. Detailed sampling procedures are found in the Barr SOPs.

VI. Study Quality Assurance

The QAPP describes the procedures and protocols intended to ensure that the data reported in the Study are of known quality and to estimate their accuracy and reliability.

A. Survey

1. *Survey Data Quality Objectives*

Data quality objectives (DQOs) are used to indicate the level of uncertainty that a decision-maker is willing to accept. DQOs are expressed both qualitatively and quantitatively. Their purpose is to ensure that the final data will be of sufficient quality for its intended use. DQOs should be determined based on the end use of the data and should also reflect limitations that exist, such as time constraints and funding limits. The development of DQOs for the Study was allowed to be iterative so that they could be updated as the Study team determined a need existed.

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[15] In series, 100 mL of aqueous solution each (except for desiccant): deionized water, sodium hydroxide, sodium borate, desiccant

The survey was sent to all processors who agreed to participate. The processors were asked to provide as much information as possible on the survey form, but surveys containing partial data were returned in nearly all cases. The processors were asked to provide raw material and product data based on product inventories that were expected to be reasonably accurate and precise. The processors did not know the amount of APFO in the purchased products. Thus, they were asked to provide data on the amounts of products containing AFD used. Also, the survey requested data on destruction and discharges.

In addition to completeness, other data quality objectives were "reasonableness" and "comparability." Reasonableness is agreement between the quantity of materials input and the sum of the amounts emitted, destroyed, and output from a process. Comparability is the extent that data from one process can be compared to other similar operations.

B. Quality Control Procedures

Barr and KHA completed a quality control review of survey data during the interviews prior to sampling. The primary quality control measures were reasonableness and comparability. Reasonableness was ascertained by comparing the quantities of APFO in the raw materials to the sum of process emissions, destruction and outputs. The quality control checks for reasonableness were performed during the pre-sampling interview, during the sampling event, and following receipt of analytical data; the results were reported in the process report.

After data were obtained from multiple similar operations, the comparability of the results was assessed. The data collected from all sources were determined to be comparable. To maintain confidentiality, the comparability results were not included in the individual processor reports.

C. Comparison of Data to Objectives

The completeness and reasonableness of survey data were evaluated together with the sampling results for each process as part of the analysis for the individual process material balances. The comparability among processes of the survey data and sampling results were evaluated as part of the aggregate analysis.

D. Data

Barr completed a data validation of the Exygen reported results for the APFO analyses of solid, liquid and air samples. Exygen performed all analyses using high pressure liquid chromatography (HPLC) with a tandem mass spectrometric detector (MS/MS). All sampling, analysis and data validation were performed according to the procedures outlined in the *Quality Assurance Project Plan, Dispersion Processor Materials Balance Project, Revision 0.0,* January 31, 2004 (QAPP) as amended by *Addendum 1.0, Quality Assurance Project Plan Dispersion Processor Materials Balance Project*, June 30, 2004.

In general, the areas covered by the validation process were as follows: analytical holding times; sample preservation and storage; mass calibration and analytical conditions; analytical instrument calibration procedures; target parameter identification criteria; method, field, and reagent blank analysis; laboratory control samples; labeled standards; matrix spikes;

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sample duplicates; target parameter quantification; data package completeness; and overall data assessment.

The data validation process involved reviewing each of the previously listed areas and either verifying compliance with the standard operating procedures in the QAPP (*i.e.* target parameter identification/quantitation, etc.) or comparing the results to the QAPP criteria (*i.e.* labeled standards/matrix spike percent recoveries). Based on the results of the verification, the data were qualified if there was non-compliance with the QAPP that could not be corrected or if the QAPP criteria were not met.

The data from the sampled processes (including a total of 428 individual analytical samples) were validated. There was good compliance with the QAPP and all criteria were met in the following areas; analytical holding times; sample preservation and storage; mass calibration and analytical conditions; analytical instrument calibration procedures; target parameter identification criteria; method and reagent blank analysis; and laboratory control samples. No corrective actions or qualification of the data were necessary based on these areas. The following paragraphs describe other areas that did not strictly comply with the QAPP or meet the QAPP criteria.

During the validation, four areas were found that did not meet QAPP criteria. These included (1) lack of field blanks collected from three of the processes sampled, (2) lack of sample duplicates collected with water samples from one process sampled, (3) several calculation errors in the target parameter quantification, and (4) several sample identification errors in the data package. Although the field blanks were not collected at three processes, an air sampling reagent blank of deionized water was collected at each process following the same procedures as a field blank. Therefore, blank data representative of the sampling conditions were available for evaluation. The samples collected without duplicate analysis data were rejected and the process was resampled to include sample duplicates. All data that were calculated or quantified incorrectly were rejected and the laboratory provided acceptable replacement data. The laboratory corrected all sample identification errors in supplemental laboratory reports.

QAPP accuracy criteria defined as spike recoveries between 70% - 130% were not met in 76 of 392 samples spiked for labeled standard spikes, and 8 of 75 solid matrix spikes. However, all labeled standard and matrix spikes were above the QAPP rejection criteria (<10%), so no data required rejection. QAPP precision criteria were not met in 4 of 71 sample duplicates.

The failure to meet the precision and accuracy criteria can be attributed to two considerations. First, the criteria were generated using deionized water blanks and were not established using real sample matrices. This may have resulted in establishing analytical criteria that are too stringent, due to the differences in the matrices of actual samples.

Second, LC/MS/MS analysis uses ionization to aid in quantifying all sample results. The ionization can be enhanced or suppressed through other constituents that may be present in the sample matrix. Based on this evaluation, samples that failed the accuracy and precision criteria were qualified as potentially biased, but were not corrected and the results were included in the analysis.

All failures to meet QAPP criteria were corrected and the majority of the quality assurance indicators showed good precision and accuracy within the analysis for most sample matrices. Given the large variety of sample media collected (solids, air impingers, wastewater) these indicators have shown that only a small percentage of the samples had any matrix effects that could have potentially biased the data. Overall the data validation procedures showed that the data are of acceptable quality to meet the project objectives.

Additional sampling data collected by outside sources were used in the Study. These outside sampling data sources represented four of the 15 processes examined in the Study. The data from these sources were reviewed to determine if the sample collection and analytical techniques used to generate the data were comparable to those in the QAPP. This review centered on the type of quality assurance samples collected, the analytical method, the quality assurance criteria (holding times, etc.) and the overall evaluation of the quality assurance data.

For two processes, it was determined that the types of quality assurance samples collected and analyzed (duplicates, spikes, and blanks), the quality assurance criteria applied, and the review of the data, were equivalent to those presented in the QAPP. A different analytical technique was used compared to the method specified in the QAPP (Gas Chromatography GC) vs. High Performance Liquid Chromatography (HPLC)); however, the quality assurance criteria for the method were met during analysis. Based on the review, the data were comparable to the sampling results for samples collected under the QAPP. For these reasons, the data from these two processes were used in this Study.

For the other two processes, sampling results were provided for wastewater, and estimates were provided for air and solid waste. Changes in personnel at the facility and the amount of time that had passed since the facility performed the sampling, prevented an evaluation of the sampling procedures. Instead, the sample results and estimates were compared to the results from other processes with similar process conditions. These results were found to be consistent with, or conservatively high compared to, results of sampling in other comparable processes. Based on this review, the data provided by these surveys collected prior to the Study were accepted for use in calculating PPFs.

Data that were obtained through surveys where sampling was not possible were also validated. For all facilities except one, validation included interviews with facility personnel, site visits, and visual inspection of the operations by DPMB team members. One facility declined to participate in the post survey phase. All data collected through surveys were further validated through a comparison to the sampling results from similar processes. The data reported in the surveys are included in this Report and are accepted as valid for those processes in those facilities. For the purpose of extending results to the industry, sampling data were given greater weight than survey data.

Process information for two-thirds of participants in this Study were obtained by on-site observations and sampling. Production schedules or other facility limitations prevented sampling for one-third of the participants included in the Study. The data collected from the surveys were comparable with the sampling results, thus, this Report includes the survey results and identifies them as "estimates by others."

E. Justification for Processes Selected and Sampled as Representative

The AFD processing industry consists of a population of large and small processors of the four product categories, including "Glass Cloth Coatings," "Additives," "Metal Coatings" (formulation and application), and "All Other." AFD processors who volunteered to participate in the material balance Study are the sample population.

In discussions with EPA before the Study was started, EPA staff raised the question of the "representativeness" of the participants in the Study compared to those who chose not to participate. To discuss EPA's question, the Study employed the following tools designed to judge representativeness including (1) sampling of more than one process for each known segment of the dispersion processing industry whenever possible; (2) sampling of processes that, in total, are believed to represent the vast majority of AFD consumption by the dispersion processing industry; and (3) using EPA databases to assess the environmental profile of the Study participants as compared to the environmental profile of the fluoropolymer industry as a whole. Based on these tools, as explained further below, it is believed that the Study results are representative of APFO-related use, destruction and releases in the dispersion processing industry as a whole.

Because of the small number of processors overall, and differences among them, it is challenging to assess which facilities are representative of the industry with respect to the uses of the AFD and, hence, where the APFO from the dispersions is likely to go. The judgmental sampling approach, described in Section III.D.4, allowed selection of appropriate representative processes for a majority of the product categories. This approach is based on EPA guidance and relies on process and facility knowledge to determine the optimal sampling design.[16] The sampled processes represent a majority of AFD consumption within the dispersion processing industry. Therefore, the sampling approach used is believed to provide representative results for the industry.

Other parameters were evaluated to further assess representativeness. Since APFO is not a regulated pollutant, no metrics are publicly available that directly relate to APFO. According to the surveys, the facilities in the sample population process a broad range of AFD, from as little as a few hundred kg to greater than 500,000 kg on an annual basis . Manufacturers report processors who use AFD in quantities containing as little as a few kilograms of APFO annually; some using as little as a few gallons a year. This illustrates that the sample population includes both large and small processors, as does the industry population. However, it is not a direct measure of a potential to release APFO to the environment.

In the absence of direct metrics, Barr used other environmental performance indicators to demonstrate that the sample population is representative of the industry population. These indicators are not specific to APFO; rather, they are overall environmental performance indicators, and they are readily available on EPA's Resource Conservation and Recovery Act Information (RCRAInfo) and Toxics Release Inventory System (TRIS) databases, both

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[16] RCRA Waste Sampling Draft Technical Guidance (8/02 EPA 530 D 02 002).

accessible through EPA's EnviroFacts Data Warehouse available through the Internet at http://www.epa.gov/enviro/index_java.html.

RCRAInfo, a national program management and inventory system about hazardous waste handlers, contains hazardous waste information. For the data retrieved in January 2005, 82% of the sampled population reported to RCRAInfo. In general, all generators, transporters, treaters, storers, and disposers of hazardous waste are required to provide information about their activities to state environmental agencies. These agencies, in turn, pass on the information to regional and national EPA offices. The regulations requiring reporting are governed by the Resource Conservation and Recovery Act (RCRA), as amended by the Hazardous and Solid Waste Amendments of 1984. The Hazardous Waste Query may be used to determine identification and location data for specific hazardous waste handlers and to find a wide range of information on treatment, storage, and disposal facilities regarding permit/closure status.

TRIS contains information on more than 650 toxic chemicals that are being used, manufactured, treated, transported, or released into the environment. For the data retrieved in January 2005, 55% of the sampled population reported to TRIS. Manufacturers of these chemicals are required to report the locations and quantities of chemicals stored on site to state and local governments. The reports are submitted to the EPA and state governments. This regulation is governed by the Emergency Planning and Community Right to Know Act (EPCRA), also known as Title III of the Superfund Amendments and Reauthorization Act (SARA) of 1986. EPA compiles these data in an online, publicly accessible national computerized database. The Toxic Releases Query may be used for basic facility information and chemical reports, which tabulate air emissions, surface water discharges, releases to land, transfers to off-site locations, and waste management activities. Waste management activities include on- and off-site recycling, energy recovery, and treatment.

Using the information in RCRAInfo and TRIS, Barr compared the facilities participating in the Study to the other facilities in the industry to assess the sample population's representativeness of the industry as a whole. The environmental indicators chosen for the representativeness evaluation include:

- RCRA status (including large quantity generator, small quantity generator, and conditionally exempt small quantity generator), and
- TRI data reporting status.

Data from the environmental indicators suggest that the sample population is representative of the AFD processing industry population as a whole. In general, if the sample population has a lesser environmental impact (*e.g.*, smaller quantity of AFD processed, fewer releases to the environment, more waste management activity, etc.) than the industry population, then the sample population would not be considered representative of the industry population. However, if the relative environmental impact of the industry population is less than or equal to that of the sample population, then the sample population would be considered a conservative estimate of environmental impact, and therefore representative of the industry population.

An AFD facility that is not regulated under RCRA is likely a small processor and likely to be processing small amounts of materials. Within the industry population, data for 49% of the facilities are available in RCRAInfo; in comparison, data for 82% of the sample population are available in RCRAInfo. These data show that the sample population includes a higher percentage of facilities regulated under RCRA, implying that the sample population may process larger quantities of materials than the industry population. By this comparison, the sample population would be considered representative of the industry population.

A facility's RCRA status is an indicator of the amount and type of waste that a facility generates. A large quantity generator (LQG) generates more waste than a small quantity generator (SQG), from which is assumed that a LQG uses more raw material and makes more product than a SQG. Within the industry population, 42% of the facilities are LQGs as opposed to SQGs. In comparison, 78% of the facilities in the sample population are LQGs as opposed to SQGs. These data imply that the sample population includes a higher percentage of large facilities, which presumably account for the majority of APFO processing. By this comparison, the sample population would be considered representative of the industry population.

Likewise, a processor that is not regulated under TRI is likely a small processor and likely to be processing small amounts of AFD. Within the industry population, data for 23% of the facilities are available in TRIS; in comparison, data for 55% of the sample population are available in TRIS. These data show that the sample population includes a higher percentage of facilities regulated under TRI, implying that the sample population may process larger amounts of materials than the industry population. By this comparison, the sample population would be considered representative of the industry population.

The above comparisons demonstrate that the industry population, having a lesser environmental impact than the sample population based on the comparison methods described, would be conservatively represented by the Study participants. Therefore, the study participants in the sample population are considered to be representative of AFD used in the industry.

The Study design called for processors to be included who processed a greater quantity of AFD. Processes with larger quantities of AFD were chosen because they are expected to have more APFO present, and therefore the samples are more likely to meet detection limits. Because some Study participants processed larger quantities of APFO than the industry, it is to be expected that more Study participants:

1) are regulated under RCRA and TRI, and
2) are LQGs.

However, small facilities could emit greater amounts of APFO to the environment than large facilities. To account for this possibility, the sample population included small processors as well. Based on this information, we believe the sample population is representative of the industry population.

TABLE 4

Evaluation of Representativeness Using US EPA's RCRAInfo and TRIS Databases

DATA ANALYSIS

RCRAInfo Data [1]

Population	% of Facilities with Data in RCRAInfo	RCRA Status	
		% LQG	% CE/SQG
Industry	49	42	58
Study Participants	82	78	22

TRIS Data [2]

Population	% of Facilities with Data in TRIS
Industry	23
Study Participants	55

NOTES

[1] RCRA data from EPA's RCRAInfo database.

[2] 2001 TRI data from EPA's TRIS database. Waste management activities include on- and off-site recycling, recovery, and treatment.

ACRONYMS

CESQG	Conditionally Exempt Small Quantity Generator
LQG	Large Quantity Generator
RCRA	Resource Conservation and Recovery Act
RCRAInfo	Resource Conservation and Recovery Act Information
SQG	Small Quantity Generator
TRI	Toxic Release Inventory
TRIS	Toxic Release Inventory System
USEPA	United States Environmental

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VII. Individual Process Descriptions

Generic process descriptions were prepared from the individual processor site reports. Included below are generalized schematics of the processes from observations made during the Study and from available sources in the public literature. The schematics do not represent any particular processor's process, and each processor has differences that make its process unique. However, the similarities for the purposes of the Study allowed the data to be aggregated across processes as described below.

Four symbols, a diamond, a square, a hexagon, and a triangle, are used on the schematics to identify the locations at which samples and/or data were obtained. The schematics are intended to give the reader a general idea of what is involved in the different kinds of processing. At first glance, the processes may appear to be highly dissimilar. The schematics make apparent the commonalities that allow extension of the sampling results and conclusions to other similar processes within the industry.

A. Glass Cloth Coating

Fiberglass cloth coated with fluoropolymers yields weather and chemical-resistant products for use in industrial and architectural applications. Industrial applications include nonstick belts that are used in continuous ovens including food processing applications, and other types of equipment. The architectural cloth is used as a roofing system in a variety of buildings. Examples include sports stadia and airport terminals. The advantages of the use of coated glass cloth in architectural applications are improved wear resistance of the coated cloth to weathering, superior strength, and an attractive long-lived appearance.

The coatings used in glass cloth coating are principally made up of AFD to which up to 60% of other materials, including water, are added. None of the processors in the Study added APFO to the products used in the Study. Therefore, all (100%) of the APFO coming into the glass cloth coating sector comes from the dispersions sold by fluoropolymer manufacturers.

The AFD used in the coatings are obtained directly from the fluoropolymer manufacturers. In many cases, the fluoropolymer manufacturers will custom design AFD at lower solids levels for specific applications. Although the addition of other ingredients is common, the specific formulations are proprietary. The dispersions are transferred into the process baths by pumping from 250 gallon totes or pouring from drums and pails.

Coating the glass cloth involves a semi-continuous process of feeding of the cloth (the "web") through a dip tank filled with dispersion, heating in an enclosed oven or tower, and winding the coated web product on a spool (Figure 1). To start the web, an uncoated web leader is threaded through the coating line. A series of rollers guide the web through the entire line, including the oven. After the line is threaded, lowering the web into the dip tank starts the coating step. The amount of coating picked up onto the glass cloth is related to the chemistry of the dispersion and the structure of the glass cloth. Rollers remove the excess coating, which drains back into the dip tank. The coated web typically travels a short distance (3 to 6 meters, or 3 to 6 yards) before entering a slot in the oven wall. The web path through the oven is typically vertical and the oven is often called a tower. Once the oven drying and sintering steps are completed, the cloth is rolled on bolts and packaged for shipment to the customer. The customers further process the cloth into the final product, such as a conveyor belt or a roof panel.

The amount of polymer picked up in the individual processes was determined during the field work and ranged from 51 g/m^2 (1.5 oz/yd^2) to 169 g/m^2 (5 oz/yd^2). Depending on the processor, the web may be from 102 cm (40 inches) to 457 cm (180 inches) wide. The batches of cloth coating are run for 3 to 6 hours in duration based on the product manufactured and the length of cloth on the roll. Typical oven exhaust rates range from 2000 to 10,000 cubic feet per minute (57 to 280 cubic meters per hour). The coatings generally are stored and applied at ambient temperature.

After the cloth passes through the bath, the coated fiberglass material is passed through heated zones of increasing temperature, typically in a tower configuration. One zone, typically operating at 110 deg C (230 deg F) to 132 deg C (270 deg F), will drive off excess water; some additives may also be released at this point. Rollers may also be used to remove excess water, sometimes with pressure to force the polymer into the fiber structure of the cloth. A second, higher temperature zone, typically 293 deg C (560 deg F) to 343 deg C (650 deg F), may be used to "fuse" the coating to the fiber. The cloth may be coated again and again, passing through the heating processes as many as eight times or more, depending on the end-use application intended.

For PTFE coatings, a final heating step to sinter the polymer onto the cloth is performed in ovens whose final temperature zone is 371 deg C (700 deg F). Greater than 90% of coated glass cloth is coated with PTFE. The sintering temperatures for other fluoropolymers are shown in Table 1. Because of the insulating properties of the polymer, the coated web temperature remains significantly cooler than the oven air temperature. Achieving final article performance properties requires that the temperature reach 342 deg C (658 deg F) for a sufficient time for all of the fluoropolymer in the article to melt. The polymer time-at-temperature thus depends on the thickness of the polymer coating applied, to develop the chemical and heat resistance characteristic of fluoropolymers. Processing time in the oven is adjusted to accomplish this.

Chemicals from the glass coating may be released in measurable quantity in the low and intermediate heating steps if the air used in drying is not treated to remove them. Ovens installed in the last 25 to 30 years are typically designed to conserve energy by recirculating the heated oven air. Recirculation increases the heat energy and residence time seen by any dispersion chemicals, including APFO, released at low and intermediate temperatures by increasing the temperature and time-at-temperature in subsequent oven zones.

Water and solid waste are generated and are thus potential sources of environmental release. Wastewater is typically generated in equipment and container cleaning operations, as there is no process water that comes in contact with the coatings or coated materials. Solid wastes consist primarily of semi-solids and dried dispersions from the raw material and coated waste cloth that is trimmed from finished product. Nearly all the trim waste will have the same characteristics as the finished product created after the final sintering stage. Minor amounts of trim waste may be produced that may not have the same characteristics as the finished product, such as material from intermediate trimming and from roll splicing activities.

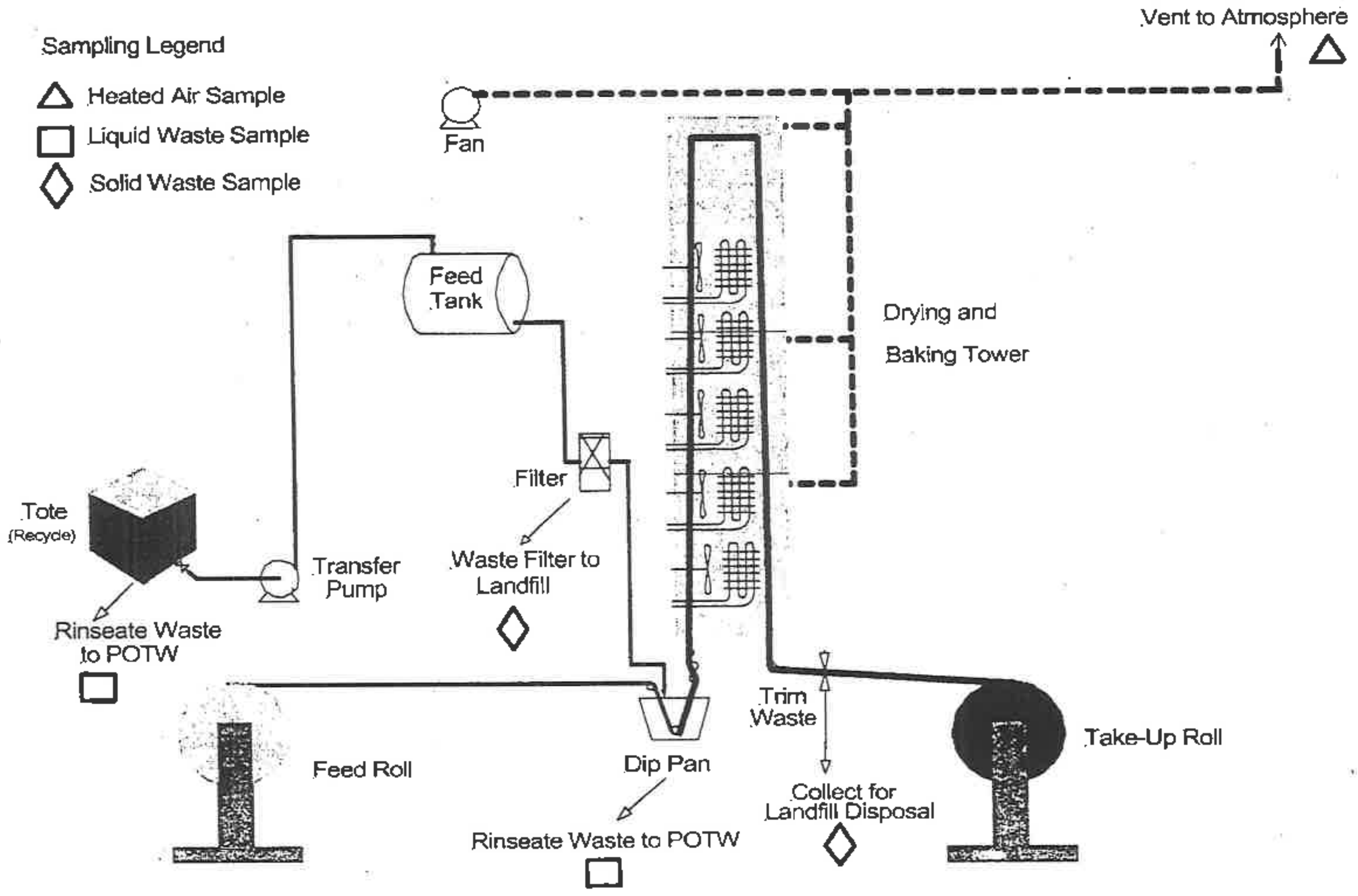

Figure 1 - Typical Glass Cloth Process

B. Manufacturing and Formulating Coating Products

Formulated coating products specifically designed for coating metal, glass, and some polymeric substrates yield properties such as wear resistance and easy-release (nonstick) characteristics. Applications include cookware, industrial equipment, and machine parts, such as for business machines. The use of these formulated coating products in industrial applications is advantageous due to the improved wear properties where close tolerances and high bearing loads in machinery would result in excessive wear. All formulated coating products must be stable mixtures allowing for handling, shipment, and use of these formulations in coating applications; hence, surfactants are essential components of the formulations.

The coating formulators typically add the AFD and other components to a mixing tank, following a recipe that specifies the amounts and order of addition of the various ingredients. In many cases, the fluoropolymer manufacturers will custom-design dispersion at lower solids for specific applications. Although the addition of other ingredients is key to the coating formulation technology, the specific formulations are CBI. None of processors contacted or observed in the Study reported the addition of any APFO to their formulations, and none of products observed in the Study contained additional amounts of APFO.

The amount of raw material dispersion used in the formulated coating product will vary depending on the characteristic of the final product. Formulations are available as coating systems, consisting of a primer coat, sometimes a mid-coat, and a top coat. Each requires different ingredients and has different properties. All aqueous fluoropolymer formulated coating products contain only AFD as the source of fluoropolymers. There are non-aqueous fluoropolymer coatings that are manufactured from other dry fluoropolymer materials (not AFD containing APFO), both solvent-based and water-reducible coatings. Neither the water-reducible nor the solvent-based products have significant amounts of APFO in the mixture because the dry fluoropolymer ingredients are heat-treated by the fluoropolymer manufacturer in normal processing prior to sale to the coating formulator.

Formulated coatings are made from AFD, to which up to 80% (based on the sampled processes) of other materials such as pigments, stabilizers, flow agents, and other additives are added. Processors reported no other sources of APFO in the formulated coatings. Therefore, all (100%) of the APFO in formulated coating products is a residual component from polymerization of fluoropolymers used in the dispersions.

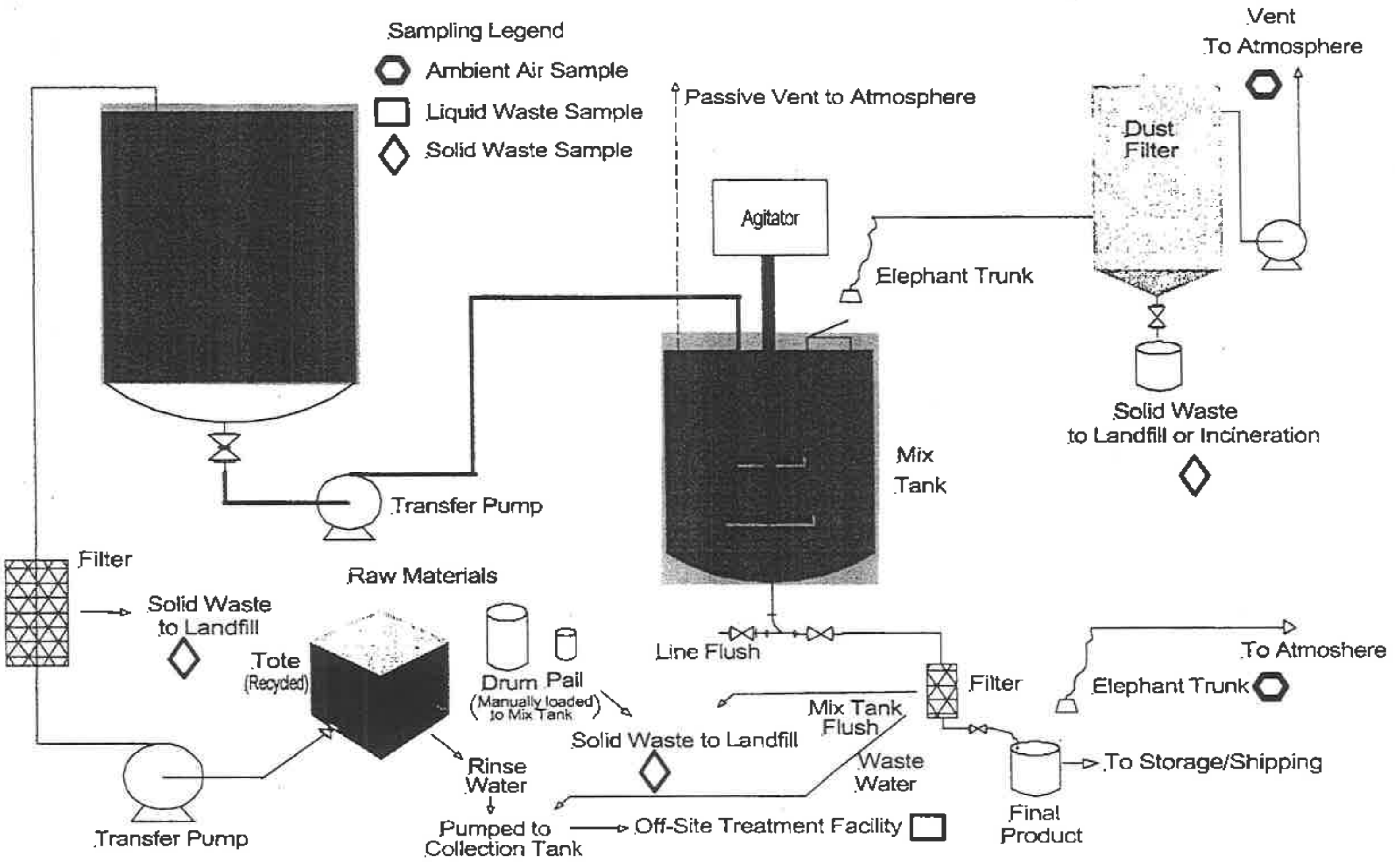

Figure 2 - Formulating Fluoropolymer Coatings

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Coating formulation batch sizes vary from 50 gallons to 1000 gallons (190 to 3800 liters). Smaller quantities can be made in laboratory-scale mixing equipment. All final formulated coatings are transferred into small containers, pails, or 250 gallon totes (950 liters) for shipment to the customers.

The incoming raw material dispersions are transferred into a mix tank by pumping from totes or pouring from drums and pails (Figure 2). The mixing tank is equipped with a slow speed agitator; high-speed mixing of dispersions can result in destabilization of the dispersion and coagulation of the fluoropolymer solids. Both dry and liquid ingredients required for the coating formulation are pumped or manually poured into the mix tank and the vessel contents are stirred for a specific period of time to ensure a proper mix.

In some cases, the contents of the mix tank may be filtered and transferred to a second tank for further addition of ingredients. The need to introduce this additional step is dependent on the nature of the ingredients and the final product properties. All mixing operations in the Study occurred at ambient temperature, with only minor increases in the temperature of the mixture in processing. This reduces the potential for APFO to be released from the mixture into the tank head space or into the workroom air.

Due to the low temperatures encountered in coating manufacturing, it was expected that releases of chemicals from the manufacture of formulated coating products that are likely to be of a measurable quantity would only occur in the cleaning and rinsing operations. Relatively small amounts of wastewater and solid waste are generated and are thus potential sources of environmental exposure. Wastewater is typically generated in equipment and container cleaning operations, as there is no process water that comes in contact with the formulated coating materials. Solid wastes consist primarily of semi-solids and dried dispersions from cleanup of the raw material. All formulators reported that wastewater and solid wastes are collected and segregated and sent for treatment and proper disposal.

C. Metal Coating

Metals and other materials coated with fluoropolymers become wear resistant, have substantial lubricity, and are useful as easy-release (nonstick) articles in a variety of industrial, commercial, and consumer applications. Industrial and commercial applications include coated parts that are used in industrial equipment and machines including business machines, automobiles, aircraft, medical devices, safety devices such as automatic pressure-relief valves for oil wells, commercial cookware and food-processing equipment, and numerous other types of equipment. The industrial coated parts also include rotating shafts and slide pins used in various machines such as farm tractors and laboratory pumps.

For consumer uses, coating of cookware is a common application for metal coating processors. For both consumer and industrial applications, the use of these coatings is advantageous due to the resistance of the coating to wear, temperature and chemical stability, and quick release or nonstick characteristics.

The metal coating processors use the formulated coating products as manufactured by formulators, an intermediate processor in the AFD industry. Coating applicators typically add nothing to the coating except occasionally water to thin or reduce the coating viscosity. In many cases, the formulated dispersion manufacturers will custom-design coating formulations for specific applications.

None of the metal coaters included in the Study added APFO to the products purchased from the formulators. Therefore, all (100%) of the APFO coming into the metal coating sector comes from the coatings containing AFD sold by coating formulators.

In the metal coating operation, formulated dispersions are transferred into a small pot by pouring from drums and pails (Figure 3). A pressurized air supply line to the top of the pot is used to force the formulated dispersion out though a feed line to a spray gun. The article to be coated is transferred to a spray booth, either manually or continuously, typically on a chain-driven conveying system. Airless spray systems have been evaluated and are not typically feasible.

The spray guns used are high-volume low-pressure (HVLP). HVLP technology enhances transfer efficiency by using a higher volume of air at lower air pressure to atomize coatings.[17] By increasing the transfer efficiency and reducing the fraction of wasted formulated coating products, this technology extends the useful life of booth filters, reduces the amount of formulated coating products lost to solid waste and increases the amount of coating applied on parts. As a result, more of the coating, and hence the APFO in the AFD is transferred to the article and thereby subject to sintering temperatures in processing.

In an automated system, to start the coating, the spray nozzle is set up and aimed at the article at the proper angle (depending on the configuration of the article, two spray nozzles may be used). Trip switches on the conveyor system activate the spray gun in sequence with the article passing under the spray nozzle, resulting in each part being coated. In a manual system, workers apply the coatings like common paint or coatings.

The spray application is always performed in a typical spray paint booth. Both walk-in and smaller bench-top booths may be used. Overspray from the coating operation is collected on filters and may also be collected on paper liners placed on the floor and walls of the spray booth.

Coating systems are typically two- and three-part systems, consisting of a primer, mid-coat, if applicable, and top-coat. On automated lines, there is frequently a low-temperature drying or baking step between coats. Some processors who have higher production rates, such as in the cookware industry, heat the freshly-coated parts using infrared heat sources, then cool them with moving air. Manual production lines may dry coated parts at ambient conditions between coats. This is called "flash-drying" in the remainder of the Report. In some cases, the top- or mid- coat may be applied to either the wet primer or mid-coat.

All coatings undergo some type of drying, baking, and sintering to allow them to adhere to the metal substrate and to achieve final performance characteristics. In the manual system, once the part is coated, the painter places the coated parts on an oven rack, truck, or tray, or on a

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[17] Binks Technical Bulletin, HVLPR-3, April 2004.

conveyor into the oven. Depending on the size of the job, when the rack is full, or at assigned times, the coated parts are placed in an oven for sintering. Automated systems typically convey the coated part immediately to next coating step or to the oven.

The amount of fluoropolymer applied to articles depends on the nature of the formulation, the coating thickness, the desired performance characteristics, and the nature of the substrate. The coating thickness ranges from 0.005 to 0.038 mm (0.0002 in to 0.0015 in) for each coating applied to the article. The usage rate of the coating will vary based on the size of the article being coated and method of preparing the article for coating. Some coaters will clean the parts using traditional metal cleaning and preparation techniques, such as acid baths and "sandblasting" with different abrasives prior to coating, depending on the durability of the substrate and the difficulty of achieving adequate adhesion of the coating. Some formulation chemistry is specifically designed for good adhesion to metal substrates without cleaning or abrasive blasting.

Following the initial primer application, the coating is often dried or baked at temperatures in the range of 49 deg C to 204 deg C (120 deg F to 400 deg F). Subsequent coats are baked or sintered at temperatures in the range of 121 deg C to 426 deg C (250 deg F to 800 deg F). Spray booth exhaust rates are typically 85 to 140 cubic meters per minute (3000 to 5000 cubic ft per minute). Typical oven exhaust rates are 14 to 110 cubic meters per minute (500 to 4000 cubic ft per minute).

In all cases, a final heating step to sinter the polymer onto the substrate is necessary to generate the adhesion and wear resistance characteristic of fluoropolymers. As with other processes, complete sintering is accomplished in ovens operating at temperatures of 393 deg C (740 deg F) to 426 deg C (800 deg F). Higher temperatures can be used to achieve greater throughput but may often result in over-heating of the polymer, resulting in thermal degradation and the generation of undesirable by-products. Again, achieving final polymer characteristics requires that the polymer temperature exceed 342 deg C (621 deg F) for a sufficient time for the fluoropolymer to melt, while minimizing higher polymer temperatures to control thermal degradation. The time-at-temperature for a particular coated part depends on the thickness of the fluoropolymer coating applied and the size, mass, and thermal transfer characteristics of the substrate.

Once the sintering step is completed, the article is usually returned to the coater's customer for its intended use (*i.e.,* installation of hardware on the article).

Releases of chemicals from AFD metal coating operations that are likely to be of measurable quantity may occur in the low and intermediate heating steps if the air used in drying is not treated to remove them. Water and solid waste are generated and are thus potential sources of environmental release. Wastewater is typically generated in cleaning operations, as there is no process water that comes in contact with the coatings or formulated coating materials. Solid wastes consist primarily of semi-solids and dried dispersions from the raw material, filters, rags used for cleaning, and any booth liner material used to catch overspray.

D. Additives

As the name implies, this is a process where the liquid dispersion is used as an additive, typically in other liquid products or solid articles. The article may or may not be a finished commercial or consumer good. Chapter 11 in the Fluoroplastics reference cited above describes a number of additives applications for fluoropolymers. Those applications described herein may incorporate AFD, but others described in Fluoroplastics may use PTFE micropowders as the source of fluoropolymer. In that event, because the fluoropolymer manufacturer processes the micropowder in a high-temperature cycle, the APFO content of those applications is likely to be low (ppm or less by weight) or non-detectable.

In almost all cases involving use of AFD, blending is likely to be performed at ambient temperatures, and some of the products made with fluoropolymers subsequently are either heated in processing or encounter high temperatures in use. Others are essentially sealed applications, for example, in alkaline batteries. For the purpose of the Study, where the process is similar to one sampled, an assessment of the suitability of extrapolation of the PPFs derived to the additive process is provided.

1. Additives: Oil

Oils and greases are manufactured with fluoropolymer additives to allow for use in environments where heat stability and chemical stability are important characteristics. These additives can incorporate different types of fluoropolymer resins, both dry and aqueous. For the liquid dispersion, the dispersion is blended at ambient temperature with the oil or grease and packaged for sale. The operation is a simple blending process at ambient temperature identical to fluoropolymer coating manufacturing or formulation. Use of these oils in internal combustion engines results in high heat exposure of the fluoropolymer in the combustion chamber of the car engine.

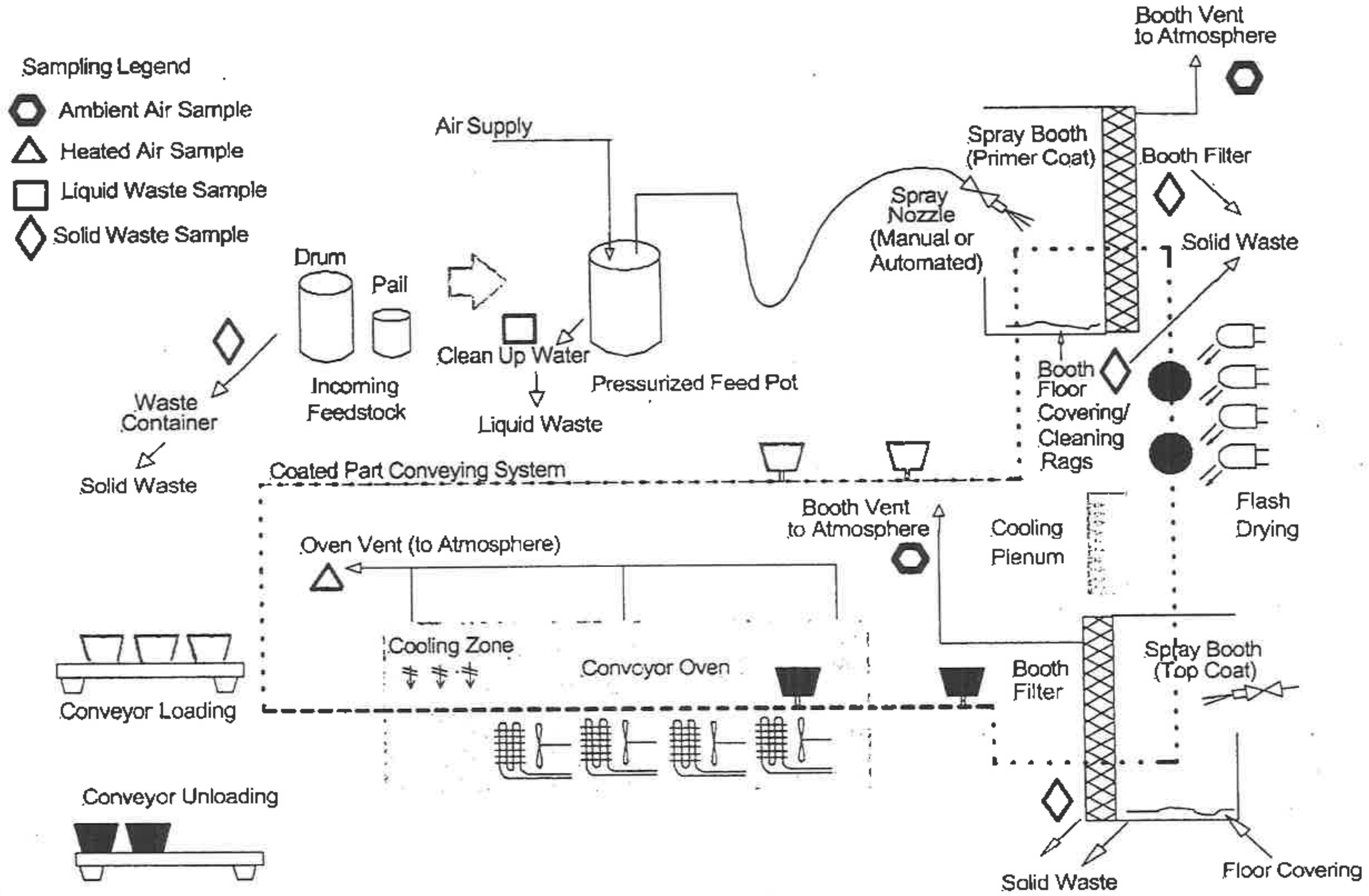

Figure 3 - Application of Metal Coatings

2. *Additives: Batteries*

AFD and other types of fluoropolymers are utilized in the manufacture of dry cell batteries. The function of the dispersion is to act as a binder for the internal battery components. Not much is known about the actual combination of the dispersion and the battery components. It is believed this operation is carried out at ambient temperatures as the other components in the battery construction will degrade at high temperatures. The operation is believed to be a blending process at ambient temperature similar to formulation, and the APFO content of potential sources of waste may be represented adequately by the PPFs derived for the coating formulation process. Because the fluoropolymer is encased in the battery, the potential for exposure to APFO from this use is negligible.

3. *Valve and Pump Packing Materials*

[IN PROGRESS]

E. All Other

The product category called "All Other" includes several types of processing to manufacture various products, including film, non-woven cloth, and fluoropolymer fibers.

1. *Cast Film and Film Coating Manufacturing*

AFD are used to manufacture and coat films with fluoropolymers.[18] Often, a combination of AFD is used to impart specific properties to the film manufactured. Applications for fluoropolymer film include communication, aerospace, electronic and optical devices. Use of these films in electronic applications is advantageous due the dielectric strength of the film.

Like the AFD used in coating formulation products, those used in film manufacture and coating are principally made up of raw dispersion to which up to 1 - 2 % of other materials, including pigments, may be added. None of the products observed in the Study contained added APFO. Therefore, all (100%) of the APFO coming into the fluoropolymer film-processing sector comes from the dispersions sold by fluoropolymer manufacturers.

The fluoropolymer manufacturers may custom design AFD at lower solids for specific applications or film coaters may add water to the dispersions to thin the coating prior to application. Dispersions are supplied in totes, drums or pails and either pumped or manually loaded into the process equipment.

The first films manufactured were "cast" onto a heated table or drum. Casting is performed by allowing the liquid to form a thin film on a heated metal base – a table or a drum. The heat would drive off the water and other constituents in the dispersion, leaving the fluoropolymer in a thin film that can be further built up or peeled off. Once the first layer of the film is dried, the second and subsequent coats of the same or other AFD are "cast" until a final,

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[18] Fluoroplastics, Chapter 11, *Fabrication and Processing of Fluoropolymer Dispersions.*

multi-layer film is produced. Newer technology uses a multi-coat process where the dispersion is dip coated on a web, typically a continuous metal sheet, passing through a dip pan, and then through an vertical oven or tower, similar to the glass cloth coating process, where the water and other constituents are removed (Figure 4). Following dipping, the coated web travels a short distance (1 meter, about 2 - 3 feet) prior to entering the tower or oven through a slot. Bars along the width of the web regulate the coating thickness as it exits the dip pan and allow excess material to flow back into the dip pan.

The web path through the tower is typically vertical. Multiple dipping and drying/baking steps may be used to produce a multi-layer film that is peeled from the web, resulting in the final product. The film is sintered like other fluoropolymer products to achieve final characteristics.

The thickness of each layer of film coating relates to the properties of the polymer used and the method of coating utilized. The film produced will typically be from 0.00127 cm to 0.5 cm (0.0005 in. to 0.2 in.) thick and up to 152.4 cm (60 in.) wide. The typical AFD usage rate in manufacture of the coated film is on the order of 0.03 to 0.2 kg/sq. meter (1 to 6 oz/sq. yard) or up to 23.7 oz/ minute (0.7 kg/min). The processing time depends on the technology used. The

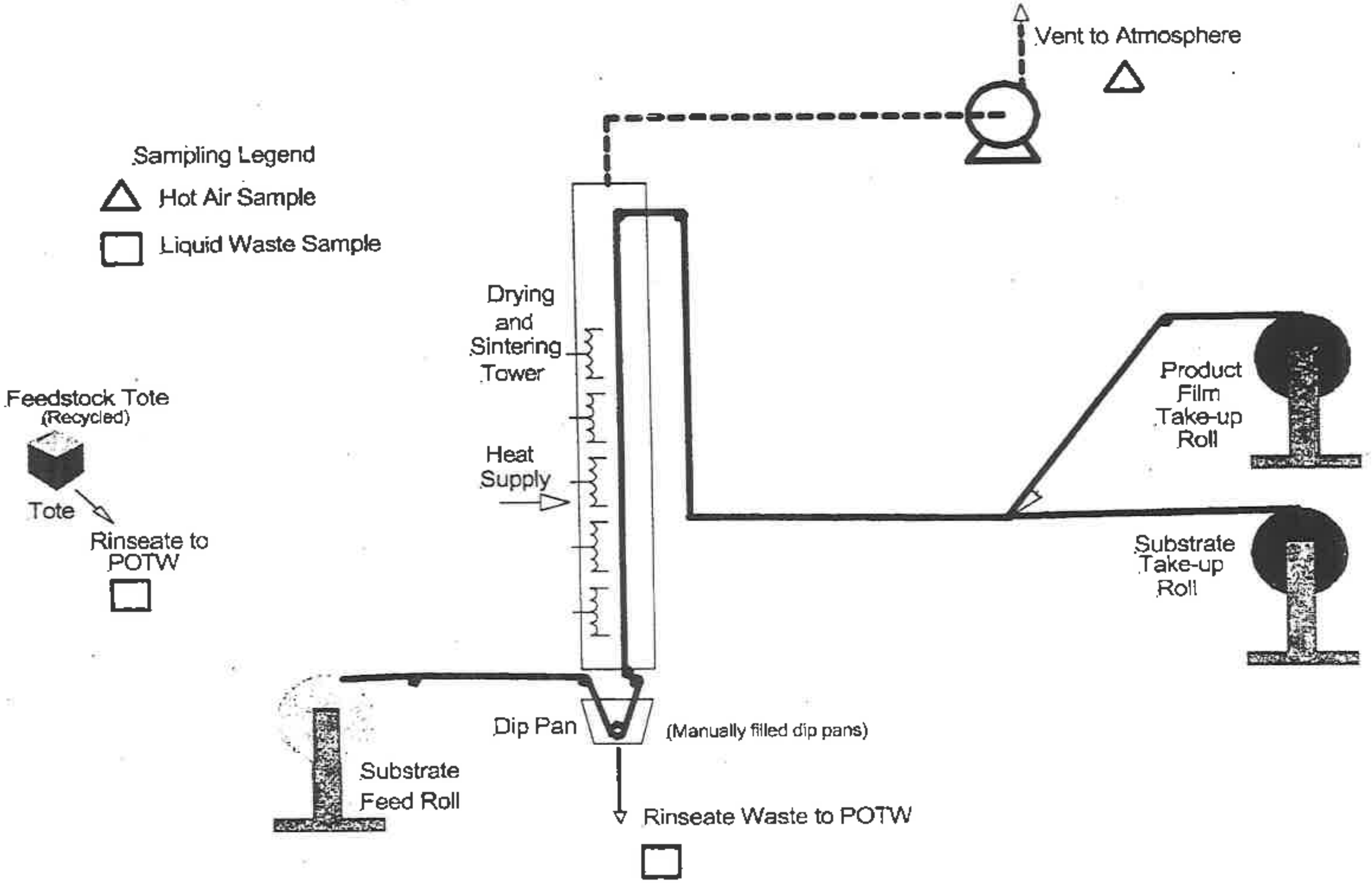

Figure 4 - Typical Coated Film Process

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total processing time can vary from three (3) to eight (8) hours depending on the width and length of the web. The coating is applied at room temperature.

Once the final film layer is applied, the film is rolled onto a roller. In some cases, there may be some final trimming of the film to specific widths for end use applications. In all cases, the film is rolled up and packaged for shipment to the customer.

Film coating involves the application of AFD to a web substrate, similar to glass cloth coating. The observed difference in this Study between glass cloth coating and the coating and heat treatment of other substrates is that the coated film is finished in radiant heat oven systems similar to those used in film manufacturing.

Releases of chemicals from fluoropolymer film manufacturing and coating that are likely to be of measurable quantity may occur in the low and intermediate heating steps, and air containing APFO could be released to the environment if the air used in drying is not treated.

Water and solid waste are generated and are thus potential sources of environmental release. Wastewater is typically generated in equipment and container cleaning operations. Solid wastes consist primarily of semi-solids and dried dispersions from the raw material and fluoropolymer film that is trimmed from finished product. The trim waste has the same characteristics as the finished product, having been processed through the same system and heating profile.

2. *Impregnated Felt Cloth*

"Felt cloth" may be manufactured from a variety of materials and substrates – such as spunbonded polyolefin fiber, aramid fiber, and others – and is used for the manufacture of filter media for air pollution control "bag houses." In cases where the incoming air stream contains a corrosive chemical, or is at a high temperature (> 300 - 400 deg C, 570 - 750 deg F), AFD are sometimes used to impregnate the felt or cloth. The fluoropolymer increases the strength of the bags.

Manufacture of these bags involves the application of AFD to a filter material, and drying but not sintering the dispersion on the substrate. The AFD is applied at a rate of 1 - 12%, giving an AFPO content of up to 0.0008%. Following coating/impregnation, the felt cloth may be heat-treated at temperatures, up to 260 - 315 deg C (500 - 600 deg F) depending on the end application and required physical properties.

3. *Fluoropolymer Fiber Production*

Fluoropolymers can be manufactured into fiber materials, which can then be woven into various products having the same properties of fluoropolymers.[19] These yarns are manufactured through an extrusion process using a spinneret, or spinning apparatus. The spinning apparatus includes a heated polymer-containing barrel, with a piston that forces the heated liquid polymer through a die. Spinning temperatures range from 220 to 410 deg C (428 to 770 deg F),

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[19] Ebnesajjad, p. 204-205, 238

depending on the fluoropolymer used. The fibers solidify as they leave the die. Common fluoropolymers used in this industry include PVDF (polyvinylidene fluoride), PFA (perfluoroalkoxy polymers), and FEP (fluorinated ethylene propylene). Fibers can be made with mixtures of PVDF with different molecular weights, but other ingredients are not typically added to the AFD as received from the manufacturers. In some product applications, fiber strength has been enhanced by adding cross-linking promoter compounds and activated by electron beam irradiation. The product is generally then reheated, stretched, and cooled in a series of steps to increase tensile strength.

Multifilament yarns consist of bundled individual filaments and are produced by an extrusion process through a spinneret die at temperatures ranging from 380 to 400 deg C (716 to 752 deg F). The die may contain 200 or more holes, each producing a single filament. The size of the individual filaments is expressed in denier, which is the gram weight of 9,000 meters of filament, or tex, which is the gram weight of 1,000 meters of filament. The filaments are then cooled using a temperature-controlled cooling chimney or by quenching through a series of water baths. Following cooling the yarn may be stretched at temperatures close to melt temperature to properly orient the molecular chains. Reorientation increases tensile strength and may reduce the diameter of the filament even further. Stretching (or drawing) can increase break elongation up to 125% and is typically done at elevated temperatures near 200 deg C (392 deg F). Once stretching has been completed, the fiber is annealed at temperatures near 170 deg C (338 deg F) to retain the properties developed from the stretching process, and wound onto spools or bobbins for subsequent use in textile operations.

Monofilament yarns consist of a single filament that is typically larger in diameter than those produced in a multifilament process. Yarn diameter is typically measured in millimeters rather than by weight, as are multifilament yarns. However, the manufacturing process is very similar to the multifilament yarn process described above, the exception being that the spinneret die has much larger holes and die temperatures are in the range of 220 to 410 deg C (428 to 770 deg F), depending on the fluoropolymer used. Monofilament yarns are typically quenched in a series of oil or water baths at temperatures near 165 deg C (329 deg F), followed by reheating to stretch and reorient the molecular chains to increase tensile strength. Stretching of monofilaments can increase break elongation up to 90%. Depending on the intended properties of the product, stretching may not be performed. Instead, the entire rolled yarn may be placed in an oven for heat treating, which may be followed by a sintering step.

Fluoropolymer fibers are used in a wide variety of woven and non-woven products. End markets for fluoropolymer-based fibers include electrical equipment, transportation, telecommunications, aircraft, and composite industries. Products manufactured include air-pollution control fabrics and filtration media for corrosive environments, gaskets and seals where chemical resistance or a low coefficient of friction is required, and electrical insulators that take advantage of the thermal stability of fluoropolymers. Consumer products based on monofilaments include protective and sports clothing, carpeting, and shoes. Fluoropolymer fiber is also used in fishing line and nets, and rope or twine, where it provides superior knotting and tensile strength.

VIII. Results

A. APFO Remaining in AFD

In 2001, the fluoropolymer manufacturers reported on total worldwide use of fluoropolymer processing aids (FPA), including APFO, and including estimates of FPA content in those products that were sold as AFD. For the DPMB project, the fluoropolymer manufacturers reported 2003 sales estimates of APFO in AFD, which are comparable to 2001 sales estimates. Participating fluoropolymer manufacturers provided APFO in AFD sales estimates for their largest customers who represented at least 90% of their total sales.

Participating processors reported total AFD or AFD products used in 2003. Based on the reported uses of AFD from the participant survey, combined with the data on the weighted average APFO content of AFD, it was estimated that the processes in the Study represent approximately 75% of APFO in AFD. The details of the data on which these conclusions are based are included in confidential attachment of this Report provided to EPA. This large percentage provides further justification for extending the PPFs generated from sampling and surveys to the remaining AFD processing industry.

B. Destruction of APFO

The measurements of the APFO content of the air, wastewater, and solid waste are used to calculate material balances for each process. The amount of APFO input to the process was determined based on each AFD manufacturer's measurements of the APFO content of its product. The amount destroyed could not be measured directly but can be estimated based on well-documented properties of APFO. The APFO content of the finished articles was not measured but was determined to be very small in most cases, based on samples of heat-treated process wastes, such as trim. The material balance equation can be solved for the sum of the amount destroyed and in the finished articles as shown below.

- $APFO_{input} = (PPF_{air} + PPF_{water} + PPF_{solid\ waste} + PPF_{product} + PPF_{destroyed}) \times APFO_{input}$

- $PPF_{air} + PPF_{water} + PPF_{solid\ waste} + PPF_{product} + PPF_{destroyed} = 1$

- $PPF_{product} + PPF_{destroyed} = 1 - (PPF_{air} + PPF_{water} + PPF_{solid\ waste})$

The thermal stability of APFO results in it being destroyed by decarboxylation when it reaches elevated temperatures for specific lengths of time. In a recently published Study, the half-life of APFO across a range of temperatures was measured.[20] Based on the data contained

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[20] Krusic, P.J. and Roe, D.C., p. 3802.

in the published Study, an equation was developed that could be used to predict the half-life of APFO for a given temperature.[21] This equation is:

$$t_{½} = \frac{\ln(2)}{T * e^{(-18.,080/T + 24.19)}}$$

where T is the temperature in degrees Kelvin. The equation can be used to predict the half-life of APFO at specific temperatures of interest in this Study. At the common sintering temperature for PTFE of 350 deg C (662 deg F), the half-life of APFO is 0.14 seconds. At this temperature, greater than 99% destruction would be achieved in seven half-lives, or approximately one second, as follows:

$$\{1-(1/2)^7\} * 100 = \{1-0.0078\} * 100 = 99.2\%, \quad > 99\% \text{ destruction}$$

7 half-lives * 0.14 seconds per half-life = 0.91 second

A similar calculation yields a half-life of 1.91 seconds at a temperature of 300 deg C (572 deg F). At this temperature, greater than 99% destruction would be achieved in 13.4 seconds.

APFO is destroyed under fluoropolymer sintering treatment. In samples of heat-treated waste material, the measured APFO content was between 1 and 150 mg APFO per kg (ppm w/w) of product. Based on the relative volumes of the waste streams, the mass of APFO in the heat-treated waste stream was negligible compared to the results from measurements in the air, wastewater, and other solid waste streams. Therefore, in processes where the polymer is heated above 350 deg C (662 deg F) for at least one second, and the temperature of the air stream above the article is at least as high, the material balance formula can be reduced and solved for the amount destroyed, as shown below.

$$PPF_{destroyed} = 1 - (PPF_{air} + PPF_{water} + PPF_{solid\ waste})$$

The published results of related studies support this conclusion. A DuPont Company Study on APFO migration from coated cookware using a Food and Drug Administration (FDA) method found no detectable levels of APFO at the reported sensitivity.[22] The results of this Study support the hypothesis that a significant amount of APFO is destroyed, based on the

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[21] Add a citation or clarification to explain who developed the equation (was it developed in published study or by us for our Study based on the published study.

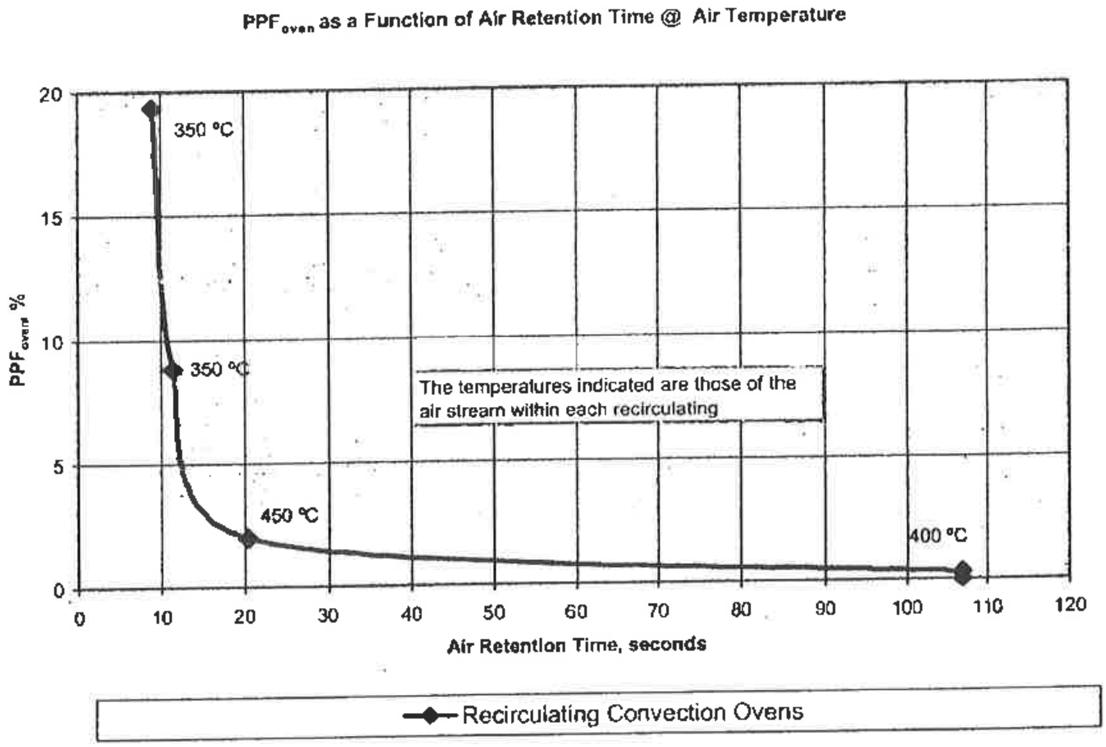
[22] Determination of Perfluorooctanoic Acid from the Surface of Commercial Frying Pans, EPA Docket AR226-1204, Analytical Report, January 16, 2003. The limit of quantitation (LOQ) was 50 ppt and the limit of detection (LOD) was approximately 10 ppt per aliquot. Expressed as a function of the surface area of the coated article, this corresponded to 100 ng/420 sq cm LOD, or 20 ng/420 sq cm LOD. The method measures the amount of a substance that can migrate from food contact items, as opposed to measuring the amount of the substance present.

[24] Perfluorochemicals: Potential Migration From Food Packaging, Begley, T.H., Presentation at the 3rd International symposium on Food Packaging, Barcelona, Spain, November 17-19, 2004. At this recent scientific meeting, FDA reported detecting very small amounts of PFOA (4-75 ppb by weight of PFOA /kg of coating) in a study in which the fluoropolymer coating was scraped from the cookware, ground up, and aggressively extracted.

thermal behavior of the compound, the sample results of the Study, and other studies in the literature.[24]

The partitioning of APFO emissions to air from the thermal heat treatment processes observed in this Study follows the behavior predicted in the literature when correlated to air retention time in the oven systems studied. APFO is likely to be destroyed when the air stream is retained for a sufficient time at air temperatures that will degrade APFO. It is thought that a majority of the APFO is released to the air stream in processing equipment from the coated articles as they are heated, and that destruction is the result of the heating of the air stream as it is circulated in the oven systems. Figure 5 below illustrates the relationship between PPF_{air} for ovens (PPF_{oven}) and air retention time in the oven at various temperatures observed in this Study. At these temperatures, longer air residence times correlate with lower oven PPFs.

Figure 5



The level of destruction appears to fall somewhat short of what would be expected based on the published predictions of APFO half-life at these temperatures. There are two possible reasons for this. First, variability in oven air temperature due to imperfect mixing would reduce the time that the air stream is subject to temperatures that will destroy APFO. Second, APFO is continuously released as freshly coated articles enter the ovens at the same time that some fraction of the oven air is vented to the atmosphere, setting up a circumstance where some APFO laden air is vented before it has been subjected to sufficient time at high temperature.

The destruction of APFO is also dependent on the type of oven used for heat treatment. Convection ovens that recirculate the air to reduce energy consumption provide both the higher air temperatures and longer air retention times that support the destruction of APFO. The air temperature in recirculating ovens is monitored as a process operating variable in the treatment of coated articles, as heat transfer is accomplished by air convection.

Non-recirculating, radiant heat ovens have relatively short air retention times and do not heat the air sufficiently to provide a similar level of APFO destruction. Radiant oven data are not shown in the above figure, as air temperature is not typically monitored in these oven systems. Rather, the surface temperature of coated articles is monitored and controlled to ensure sufficient radiant heat transfer to produce finished articles.

C. Aggregation Across Processes

As expected, the results from sampling processes within a market segment were comparable. A high degree of comparability was found among processes from multiple market segments as well. Most remarkably, the APFO levels detected in wastewaters, largely generated by container and process equipment rinses, were highly comparable. All rinses were made using water at ambient temperature. The APFO detected in wastewaters where there was no direct contact of process water with the dispersion-treated product ranged from 0 to 2.8% of the APFO input.

One facility disposed of all remaining unused dispersion at the completion of a production run by discharging the remaining dispersion to an on-site wastewater treatment plant. It was determined that this practice was not typical of any other processor and the resultant PPF calculated for this facility was not representative of any other sampled or surveyed facilities. To provide a comparison with PPFs calculated for other process wastewaters, this PPF was divided into two PPFs that (1) represented the amount of APFO attributable to only dispersion disposal and (2) represented only the amount of APFO lost to process rinseate. The PPF that represented only the process rinseate fell within the range typically found for process wastewater for all other facilities, consistent with results for all other process wastewaters.

Where the dispersion-treated product was in direct contact with process water, the process water contained up to 22% of the amount of APFO in the AFD used. Most processes used water only for cleaning containers and process equipment. Therefore, it would be appropriate to conclude that for most processes, the PPF_{water} is 3% because water is used solely for cleaning. The PPF_{water} for the few processes with other uses for water would be higher (up to 22%) to include those other uses.

Similarly, the APFO detected in solid wastes was comparable for all processes in multiple market segments, with the exception of spray coating and certain processes in the "All Other" category. The APFO detected in solid wastes for all sampled processes except for those in these two categories was less than 1%. In these other processes, the APFO detected in solid waste ranged from 9 to 18% of the APFO input. Therefore, one conclusion of the Study is that the PPF_{solids} for processes in multiple market segments other than spray coating and the "All Other" category is less than 1%. A second conclusion is that the PPF_{solid} for spray coating processes and certain processes in the "All Other" category is less than 16% of the APFO in AFD.

The air sampling results also can be aggregated across processes in multiple market segments when the processing and oven type is taken into consideration. The APFO detected in air samples for process steps conducted at ambient temperature, to 65 deg C, was less than one percent of the total input APFO. At processing temperatures between 300 and 400 deg C, the APFO detected in air from recirculating ovens was 19% or less, and in air from non-recirculating or radiant ovens was approximately 40 - 54%. For temperatures above 400 deg C, the APFO detected in air was 2% or less. Thus, the results of the Study suggest the PPF_{air} is comparable across market segments when the processing temperature and oven types are considered. Therefore, for low-temperature and high-temperature recirculating-oven processes, the PPF_{air} is 2% or less. For ovens that do not recirculate the air or operate at temperatures between 300 and 400 deg C (572 - 752 deg F), the PPF_{air} can be estimated at 54% and 19% respectively.

D. Results of Sampling and Surveys by Process

The following sections summarize and discuss the derivation of PPFs by process. In the analysis by process, the sampling results for release points within an environmental medium are summed to produce a single PPF for that environmental medium. For example, wastewater containing APFO at most facilities is derived from two sources, rinsing containers that held the dispersion feedstock and rinsing the process equipment used to make the finished product. These samples are identified as "container rinseate" and "process equipment rinseate," respectively. The concentration of APFO in the wastewater from these individual samples was analyzed and results in a PPF (percentage) of the total APFO used in that particular process. In order to derive the PPF for water from all release points for the entire process, the PPFs of the individual samples were added together. For example, if two container rinseate samples and one process equipment rinseate sample were collected and analyzed from one process, the PPFs from those individual samples were added to yield the PPF_{water}. Thus, for those processes in which the only source of wastewater is from container and equipment cleaning, a PPF based on the combined results from all processes can be used. The same concept was applied to the air and solid waste streams.

Where more than one process was sampled in a process category, a range of PPFs is presented for the sampled media. This range represents the PPFs calculated for each individual process. However, to present a single PPF value for each media in each process category, the higher end of the range was selected as a conservative estimate for that PPF. This conservative estimate is then used as the PPF for that media in that process category for comparisons or calculations made later in the report.

In describing the conclusions in the Study, the word "conservative" is used to describe PPFs that are derived from direct measurements. The word "reasonable" is used to describe PPFs that are derived indirectly by subtraction and from data other than direct measurements.

In some cases, the amounts measured are such a small fraction of the input APFO, it is reasonable to conclude that the PPF for that process and medium is negligible.

Because the Study does not include direct measurements of the amount of APFO destroyed or remaining in the product, the PPFs for these values are deduced from other data. It is known that APFO degrades rapidly at temperatures in excess of 350 deg C (662 deg F). In the absence of energy or mechanisms causing the APFO material to become airborne,and the AFD remains at ambient temperatures throughout the process and does not appear in the environmental media, there is no reason to believe the APFO will not remain in the product. Using this approach, we are able to provide a reasonable estimate for $PPF_{product}$ and $PPF_{destroyed}$ for many of the processes. In other cases, the measured PPF is so small that it is negligible in the calculations.

1. ~~Glass Cloth Coating~~Glass Cloth Processing

As described previously, a continuous web of glass cloth is fed through a dip tank to apply the coating and the web is heated to dry and sinter the finished article. The only significant potential air stream is the oven exhaust. Samples at this release point were collected. Wastewaters are generated from rinses of the raw material containers and rinses of the process equipment, such as the dip pans, and samples of all wastewaters were collected. Samples of solid wastes included raw material filters and "trim" wastes from heat-treated finished product.

The APFO detected in the air exhaust from the oven ranges from 9 - 19% for sampled processes. One facility participating by survey estimated that approximately 2% of the APFO is present in the oven exhaust, but sampling data were used to establish the PPF for this process. The oven air temperature favors destruction of APFO at the process conditions. A greater level of destruction would be expected at the observed oven temperatures; however, it is reasonable that the oven air may not be perfectly mixed, so some of the exhaust air may not have reached the overall oven temperature. The range in the results from the sampled glass cloth processes is comparable. As a result, a conservative estimate for the PPF_{air} is 19%.

The APFO detected in water from glass cloth coating is limited to container and process equipment rinses. The volume of water generated is relatively small, and a small fraction of the APFO input to the process is detected in the wastewater, ranging from 1 to 3%. Therefore, a conservative estimate for the PPF_{water} is less than 3%. The wastewater generated is not discharged directly to any surface water body, but rather is treated either on site or off site.

The solid waste from glass cloth coating consists primarily of raw material filters and heat-treated product waste. The solid wastes generated by glass cloth coaters are sent off site to be incinerated or are managed in controlled landfills. The APFO detected in solid wastes is less than 1% of the APFO input. As a result, a conservative estimate for the PPF_{solid} is less than 1%.

The APFO remaining in the product can be predicted to be very small from the observations in the Study. The APFO detected in samples of the waste from heat-treated glass cloth product was less than 1 ppm by weight. The measured amount is negligible compared to the amounts detected in other media. Therefore, a reasonable estimate for the $\text{PPF}_{\text{product}}$ is zero, or negligible.

A significant amount of the APFO input to the glass cloth process is destroyed. Recirculating ovens are typically used with an oven air temperature between 300 deg C (572 deg F) and 400 deg C (752 deg F). Therefore, the process conditions are such that the APFO not detected in other media is likely destroyed. Trim waste subject to the same heat treatment as the product had less than 0.1% APFO content, and it follows that the product is likely to have the same APFO content as the trim waste. Thus, because more than 78% of the APFO input was not detected in environmental media or in product, a reasonable estimate of the $\text{PPF}_{\text{destroyed}}$ is 78%.

The above PPFs for the various media sum to more than 100 percent. This is a common occurrence for process categories as typically more than one process was sampled within each category. This occurs because each PPF range represents more than one sampled process for this process category, and, as stated above, conservative assignments were made within the PPF range for each media. By using this conservative approach, the PPF for one media may come from one sampled process while others come from a different sampled process within the same market segment. Thus when they are summed, the result can be greater than 100%. However, we can account for this by normalizing the results to 100%. To normalize, we simply add the PPFs (19 + 3 + 1 + 78 = 101), and if the sum exceeds 100%, multiply the individual PPFs by the ratio of 100 divided by that sum (multiply by 100/101). If normalized, the PPFs would be approximately 77%, 19%, 3%, and 1% for destroyed, air, water, and solids, respectively.

2. *~~Formulation~~ Formulating ~~of Metal~~ Coatings Products*

The coating formulation process consists of slow-speed blending of AFD with pigments and other ingredients at ambient-to-slightly-elevated temperatures. The only air source is the tank vent, so that based on the physical and chemical characteristics of APFO and AFD, one would predict air streams from this process to contain negligible amounts of APFO. Wastewaters are generated from rinses of the raw material containers and rinses of the process tanks, but there is no process water in contact with the AFD treated product. Samples of all wastewaters were collected. Samples of solid wastes included raw material filters.

At the process conditions for formulation, the APFO detected in the air exhaust was expected to be very small. The quantity was measured to be <0.0001% of the APFO input. This result makes sense given the low temperature and the small volume of air exhausted from the process tanks, and the absence of any process that would cause the APFO-containing mixture to become airborne. As a result, a conservative estimate for the PPF_{air} is zero, or negligible.

The APFO from the input AFD detected in water from formulation is limited to container and process tank rinses. The volume of water generated is relatively small, and a small fraction of the APFO input to the process is detected in the wastewater, less than 1%. For formulation, a conservative estimate for the $\text{PPF}_{\text{water}}$ is less than 1%. The wastewater generated is not discharged directly to any surface water body but rather is shipped off site for incineration or other water treatment.

The solid waste from formulation consists primarily of filters and process waste. The solid wastes generated by formulators are incinerated off site or are managed in controlled landfills. The APFO detected in solid wastes is less than 1% of the APFO input. As a result, a conservative estimate for the PPF_{solid} is less than 1%.

The process conditions for formulation do not reach temperatures that would destroy APFO. Thus, the APFO is carried through the formulation process and remains in the product. Destruction of APFO may occur in the downstream AFD metal-coating process. For formulation, a reasonable estimate for the $PPF_{destroyed}$ is zero, or negligible.

Nearly all of the APFO in AFD is expected to remain in the formulated coating product. The only APFO expected to be lost from the formulated coating products is the small amount that was detected in the water and solid wastes generated. Therefore, the $PPF_{product}$ is greater than 99%. As discussed below, as a result of downstream processing by the processors who use formulated coating products in their facilities, little APFO remains in the final consumer products.

3. *Metal Coating*

As described previously, the metal coating process consists of manual or automated spray coating in a spray booth. Ambient-temperature drying or a flash-drying step at approximately 65 deg C (149 deg F) may be used for multi-coat systems. The articles are heated at temperatures greater than 400 deg C (752 deg F) to dry and sinter the coating as the last step in all metal coating operations. The air release points include the spray booth and the oven exhausts. Air losses due to ambient or flash drying were evaluated and determined to be insignificant as a fraction of the APFO in the input AFD. Wastewaters are generated from rinses of the raw material containers and rinses of the process equipment, such as the spray guns and pressure pots. Samples of all wastewaters were collected. Samples of solid wastes included spray-booth filters and liners.

The APFO detected in the air exhaust from the spray booths ranged from 0.7 to 6% of the APFO input. The flash drying evaluation concluded that the amount of APFO lost during flash drying was negligible.[23] The APFO detected in the oven exhaust ranged from 0.05 to 2%. The oven air temperature favors destruction of APFO at the process conditions. Based on the maximum measured results for spray booths and oven exhausts for any sampled process, a conservative estimate for the PPF_{air} for the metal coating process is 6%.

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[23] The flash drying evaluation showed that the amount of APFO lost to air during flash drying was negligible. To account for variability in the amount of coating applied, multiple samples of coated products were collected before and after each flash drying step and APFO content was determined separately.

The resulting data were evaluated by comparing averages and ranges pre-and post drying. Based on this ~~rough~~ analysis, it was concluded that the differences in APFO content between samples collected before and after the flash dry step were not substantially different. This ~~meaning~~ means that evaporation from the surface of the coated part during flash drying did not result in release of APFO into the workroom atmosphere and is not likely to be a significant source of fugitive emissions. This was true even where the samples were heated slightly (to approximately 65 deg C or 125 deg F).

The APFO detected in wastewater from metal coating is limited to container and process equipment rinses. The volume of water generated is relatively small, and a small fraction of the APFO input to the process is detected in the wastewater, typically less than 1%. The wastewater generated is not discharged directly to any surface water body but rather is treated off site by incineration or other water treatment. For metal coating, a conservative estimate of PPF_{water} is less than 1%.

The solid waste from metal coating consists primarily of spray-booth filters, liners, and heat-treated product waste. The solid wastes generated by metal coaters are sent offsite to be incinerated or are managed in controlled landfills. The APFO detected in solid wastes ranged from 9 to 18% of the APFO input and was not heat-treated. As a result, a conservative estimate for the PPF_{solid} is 18%.

A significant amount of the APFO input to the metal coating process is destroyed. Recirculating ovens are used and the air temperature in the ovens is greater than 400 deg C (752 deg F); therefore, the process conditions are such that the APFO not detected in other media is likely to be destroyed. The APFO not found in the environmental media ranged from 77 to greater than 90%, so a reasonable estimate for the $PPF_{destroyed} + PPF_{product}$ is 77%.

The above PPFs for the various media sum to 102 percent. When normalized to a total of 100% as described previously, the PPFs are approximately 75%, 6%, 1%, and 18% for destroyed, air, water, and solids, respectively.

4. *Additives: Oil and Batteries*

The additive applications that were not sampled or surveyed but for which estimates of APFO in AFD were derived include batteries and oil and grease formulations. In all cases, it appears that the products are not heat-treated before they are finished by the fluoropolymer processor. This means that the APFO in the AFD used is most likely to stay with the final product, based on comparisons to the metal coating formulator processes studied. To the extent that there are solid or liquid wastes generated, the amount of APFO in those wastes will be proportional to the amount of non-AFD in the waste. Hence, in all cases, it is reasonable to conclude that the APFO content of the various waste streams will be less than 0.3%, because APFO in the AFD is below this amount.

For batteries, the AFD are used on the internal parts of alkaline dry cell batteries. These are sealed units, which consequently is likely to result in little or no exposure to the end user. The total amount of AFD used in batteries is a small fraction of the total AFD produced.

~~Oil and grease formulations are made with small amounts of AFD and are generally used in industrial and commercial products. In consumer products, the applications are such that general consumer exposure is not likely to occur. Moreover, the amounts of AFD in such products are small, less than [illegible]%, and residual APFO from input AFD in these products is proportionally lower. In this case as well, the total amount of AFD used in oil and grease applications is a small frac~~tion of the total AFD produced.

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5. *Additives: Valve and Pump Packing Materials*

[IN PROGRESS]

6. *All Other: Cast Film and Film Coating Manufacturing*

As described previously, a continuous web of film is formed or coated on a substrate. No processors were observed using the older method of pouring the dispersion on a heated metal drum. The web is heated to dry and sinter the finished film. The only significant air release point is the oven exhaust, and samples at this release point were collected. Wastewaters are generated from rinses of the raw material containers and rinses of the process equipment. Samples of all wastewaters were collected. Samples of solid wastes included raw-material filters and heat-treated trim wastes.

The APFO detected in the air exhaust from the ovens ranged from 39 to 54% for sampled processes. The air temperature favors some destruction of APFO at the process conditions. A greater level of destruction would be expected at the observed temperatures; however, because of the radiant heating method used, the oven air may not be as hot as the product temperature and may not be perfectly mixed. This would account for the increased proportion of APFO found in the exhaust air observed. Thus, non-recirculating ovens may not produce the same level of thermal destruction of APFO as recirculating ovens.

In addition, the observed levels of APFO in the air stream were higher when the amount of AFD used (or throughput of AFD) was greater. When air testing results for each of two similar film-coating process were evaluated independently, the air PPF for the high production product was approximately 40%, whereas the PPF for the high application rate was approximately 54%. The PPF determined to be the most representative for the process was a weighted average of the two conditions. The weighting was based on the relative production volume and was based on 2/3 high production and 1/3 high application. The weighted average resulted in an air PPF of 44.5%. Given that actual processing conditions are likely to vary somewhat, it seems appropriate to choose an average of the measured levels to represent the non-recirculating oven PPF_{air}.

The APFO detected in water from cast film and film coating is limited to container and process equipment rinses. The volume of water generated is relatively small. The APFO detected in the wastewater ranged from negligible to 3% of the total APFO input to the process. This range does not include bath dispersion waste, which one facility treated as a wastewater. Disposal of remaining bath dispersion via wastewater is an unusual situation and is not representative of typical wastewater generation for other processes. The wastewater generated is not discharged directly to any surface water body but rather is treated on site or off site using incineration or other water treatment methods. For cast film and film coating, a reasonable estimate for the PPF_{water} is 3%.

The solid waste from cast film and film coating consists primarily of filters and heat-treated product waste. The solid wastes generated by cast film and film coaters are sent off site to be incinerated or are managed in controlled landfills. The APFO detected in solid wastes is much less than 1% of the APFO input. As a result, a conservative estimate for the PPF_{solid} is less than 1%.

The APFO remaining in the product is predicted to be very small. The APFO detected in samples of heat-treated waste coated or cast film product was less than 150 ppm. The measured mass of APFO in the heat-treated waste coated or cast film product is negligible compared to the amounts detected in other media. Therefore, it is reasonable to conclude that the $PPF_{product}$ is zero, or negligible.

A large amount of the APFO input to both the cast-film and film-coating processes is destroyed. The ovens typically use radiant heat and do not recirculate the air. The web reached a temperature in the ovens between 290 and 400 deg C, with oven air temperatures ranging from about 100 deg C (212 deg F) to 300 deg C (572 deg F). The process conditions are such that the APFO not detected in other media is estimated to be destroyed. Approximately 50% of the APFO input was not found in other environmental media, so a reasonable estimate of the $PPF_{destroyed}$ is approximately 50%.

E. <u>The Material Balance for AFD</u>

The PPFs discussed in Sections VIII.D.1. through VIII.D.5. represent several different calculations. Some are the PPF for an individual type of release point, some are the PPFs for a specific medium (air, water, solids) derived from combined PPFs of individual release points, and some are PPFs of a specific medium from processes supplied by survey data (*i.e.*, those facilities that provided their own data). The PPFs from the individual release points from all sources of a specific medium can be combined to yield a PPF for that particular media.

When the data were viewed in aggregate and presented as PPFs for individual release points, some obvious patterns emerged. Air results showed a dependence on the "time-at-temperature" profile of the individual release. Wastewater results depended on whether or not wastewater was produced only as a result of container and equipment cleaning, or if water was used for another purpose in the process, such as cooling or carrying the product through the process, or for emission control. Solid waste results depended on whether or not the process included spray coating and if the solid waste was heat-treated. The following sections summarize and discuss the PPFs by air, wastewater, and solid waste media.

1. Consolidated PPFs for APFO in Air

It is possible to characterize different APFO-containing air sources in the Study by the temperature of the environment, in an oven or otherwise, and the time that the product spends at the desired temperature in the process. Table 4 contains a summary of sample data on the amount of APFO in the air sources by the highest temperature environment in which the fluoropolymers are processed.

Table 5
APFO in Air Sources

Temperature Profile	PPF Range of Individual Sample Results Obtained, %	Max Observed PPF for Process Category (%)*	Max PPF of Total APFO from Estimates by Others, %
Ambient; formulators	negl – 0.00005	0.00005	NA
Ambient; spray applications	0.7 - 4.3	5.6	NA
100 – 300 C, long air residence times (minutes)	0.05 - 0.3	0.3	NA
300 – 400 C, non-recirculating ovens; short air residence times (seconds)	39 – 54	54	17.5
300 – 400 C recirculating ovens	2.0 - 19	19	2.0
>400 C recirculating ovens	0.29 - 2.0	2.0	NA

* For process categories that include multiple individual sample results, the maximum for the category is the sum of all individual sample results included in the category. Therefore, the maximum observed PPF for a process category may be greater than the maximum individual sample result.

Several observations and conclusions become apparent when the results for air sources are viewed from the perspective of the temperature profile of the process categories. Notable observations include the following:

- Air from ambient-temperature process steps contains up to 4.3% of the total APFO input to the processes (the range is between negligible and 4.3%). Ambient process steps include mixing/formulation and dip or spray coating.
- Longer air residence times for both recirculating and non-recirculating ovens generally result in greater thermal destruction and lower air PPFs.
- Non-recirculating ovens have shorter air residence time (reduced time at temperature) and correspondingly higher PPFs. The range of observed air PPFs for non-recirculating ovens is 39 - 54%.
- In general, convection ovens have longer air residence times than non-recirculating ovens and exhibit significantly lower air PPFs. The range of observed air PPFs for convection/recirculating ovens is 0.05 - 19%.
- The maximum PPF for the ambient-temperature non-spray processes sampled was 0.00005%, which suggests that less than 0.01% of the APFO used in AFD might find its way into air from these processes.
- The PPF for sampled facilities using non-recirculating ovens is approximately 39 - 54%, while those with recirculating ovens were in the range of 9 - 19%. The maximum PPF for sampled facilities that used a recirculating oven or incorporated a thermal oxidizer was 19%. The PPF for facilities (based on survey data) with recirculating ovens operating between 300 and 400 deg C is 2.0%.

- Within the surveyed processes, the proportion processed was approximately 60% in recirculating ovens and 40% in non-recirculating ovens.
  - Of all APFO in AFD, 85% is processed at temperatures at or above 300 deg C (572 deg F). It is not apparent from the information available what proportion of AFD in the industry might be processed in a non-recirculating oven with no thermal treatment of the stack gas.
  - Conservatively applying the available information to the quantity of APFO in AFD processed at high temperatures, combined with the quantity processed in recirculation ovens, and applying the highest PPF for recirculating ovens, 9.7%[24] of the APFO in the AFD from high-temperature processing might find its way into the air stream from the recirculating oven processes. Similarly, 18 % of the APFO in the AFD from high-temperature processing might find its way into the air from the non-recirculating oven processes.

2. *Consolidated PPFs for APFO in Wastewater*

The following observations characterize APFO-containing wastewater emissions in the Study. Table 5 contains a summary of the sample data on APFO in the wastewater by wastewater source.

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[24] This value is calculated by multiplying the fraction in recirculating ovens, times the amount of APFO in AFD processed in these types of ovens times the PPF for air for these ovens. Hence, 0.6*0.85*0.19 = approximately 9.7%. Similarly for non-recirculating ovens the values to be multiplied are 0.4*0.85*0.54.

Table 6
APFO in Wastewater
(Based on Samples and Estimates)

Process Description	PPF Range of Individual Sample Results, %	Max Observed PPF for Process Category (%) *	PPF from Estimates by Others, %
Container Rinseate	negl - 2.1	See combined	NA
Process Equipment Rinseate	negl – 1.0	See combined	NA
Combined Container Rinseate and Process Equipment Rinseate from Samples		2.8	
Contact Process Water	NA	NA	0.3 - 22
Dispersion Bath Waste**	5.1	5.1	NA

* For process categories that include multiple individual sample results, the maximum for the category is the sum of all individual sample results included in the category. Therefore, the maximum observed PPF for a process category may be greater than the maximum individual sample result.

** One facility disposed of remaining AFD at the completion of a production run by discharging all remaining dispersion to an on-site wastewater treatment plant. This represents a total PPF that includes APFO released in process rinseate and dispersion disposal. See below for a complete explanation.

Several observations and conclusions become apparent when the results for wastewater sources are viewed from the perspective of the unit operation included in the process category. Notable observations include the following:

- All wastewater sources are generated at ambient temperature and are not treated at elevated temperatures at the site of generation.

- APFO content in individual wastewater samples from all sources is consistently below 2.1% of the APFO input to the processes. The range of APFO content in wastewater sources is between negligible and 2.1%. Wastewater sources include container rinses and process equipment rinses.

- The APFO content in container rinses and process equipment rinses was compared. Container rinses ranged from negligible to 2.1%. Process equipment rinses ranged from negligible to 1.0%. Based on the significant overlap in the results, the differences between container rinses and process equipment rinses can be regarded as negligible. Conservatively, it is appropriate to group data from both container and process equipment rinses to calculate the PPF for wastewater. Aggregating the data in this manner yields a consolidated PPF_{water} of 2.1%.

- The total PPF_{water} for wastewater in AFD processing from all sampled process categories is 3% after combining PPFs for container rinses with those of other process rinse water sources. The calculated PPF for wastewater in coating formulation and nonmetal coating processes ranged from 0.5 to 3%. In metal coating, additive, and other processes, the PPF_{water} for wastewater ranged from 0 to 0.7%.

- The estimated PPF for processes not sampled ranged from 0.3 to 22%. The estimated PPF of 22% is from one surveyed facility where a wet scrubber is used for particulate control and process water comes in direct contact with the AFD-treated product. The use of a wet scrubber is not common among the surveyed and sampled facilities. The use of process water that directly contacts the AFD is also unusual. The combination of wet scrubbing and direct contact process water result in a larger amount of APFO in wastewater than was observed at any other processes in the Study and warrants assigning a separate PPF_{water} for such processes.
- One facility disposed of remaining AFD at the completion of a production run by discharging the remaining dispersion to an onsite wastewater treatment plant. It was determined that this practice was not typical of any other sampled facility and skewed the results as presented in the table above. The PPF_{water} in the table for this facility was divided into one PPF that represented the amount of APFO attributable to only dispersion waste disposal and another PPF that represented only the amount of APFO lost to process rinseate. The PPF that represented only the process rinseate was found to fall within the range typically found for process wastewater for all other facilities.
- The total amount of AFD used in those processes with higher water PPFs is less than 20% of the total APFO in the industry. Thus, the wastewater stream from this process has a small impact on the industry-wide value of APFO contained in wastewater from the processing of AFD.

3. Consolidated PPFs for APFO in Solid Waste

The following observations characterize APFO-containing solid wastes sampled in the Study. Table 6 contains a summary of sample data on the amount of APFO in the solid wastes by process.

Table 7
APFO in Solid Waste Samples

Process Category	PPF Range of Individual Sample Results, %	Max Observed PPF for Process Category (%) *	PPF from Estimates by Others, %
Spray Coating	negl – 10	18	NA
Non-spray Coating	negl – 0.19	0.2	NA
Formulators	negl – 0.12	0.12	NA
Other	NA	NA	13

* For process categories that include multiple individual sample results, the maximum for the category is the sum of all individual sample results included in the category. Therefore, the maximum observed PPF for a process category may be greater than the maximum individual sample result.

Several observations and conclusions become apparent when the results for solid waste sources are viewed from the perspective of the unit operation included in the process category. Notable observations include the following:

- Solid waste samples consisted of container wastes, clean-up rags, gloves, tape, raw material filters, trim waste (of heat-treated product), spray-booth liners, and spray-booth filters.
- All solid waste streams are subject only to ambient temperatures, with the exception of trim waste, which is subject to the same heat-treatment time and temperatures as the products being coated.
- Heat-treated trim wastes account for up to 0.2% of the total APFO input to the processes.
- The APFO solid-waste sources from spray coating operations account for 18% of the total APFO input to the spray coating processes. These solid wastes include spray-booth filters and paper booth-liners that capture overspray associated with spray coating operations and understandably have a greater amount of fluoropolymer on them.
- Estimates of APFO in solid wastes from unsampled, non-spray processes can account for up to 13% of the total input APFO.
- For the remaining processes, the combined solid-waste samples generally account for up to 0.2% of the total APFO input to the processes (individual sample range is negligible to 0.19%).

The maximum PPF for solids in AFD from any process category, sampled and estimated, is 18% and the minimum is negligible.

4. *Consolidated PPFs for All Media*

When the PPFs for each process category are multiplied by the quantity of AFD used annually by each category, the results can be summed by environmental media to produce a consolidated PPF for the entire industry. An overall sum for a given environmental medium was calculated. The highest PPF was chosen from the available results for each market segment and was multiplied by the annual volume of AFD consumed by that market segment to obtain the estimated amount of APFO in an environmental medium from a single market segment. The result for each market segment was then summed for that environmental medium to represent the total amount of APFO found in that medium. To calculate the percentage of APFO in a medium, that total was divided by the total input amount of APFO in AFD and multiplied by 100.

The results are as shown below:

- PPF_{water} 5 (measured);
- PPF_{waste} 5 (measured);
- PPF_{air} 16 (measured);
- $PPF_{destroyed}$ 62 (estimated); and
- $PPF_{undetermined}$ 12 (estimated).

The PPFs for the environmental media are based on measurements. The $PPF_{destroyed}$ is an estimate that is largely based on measurements of heat-treated surrogates for products (trim wastes). To close the material balance, the remainder is called $PPF_{undetermined}$. The remainder represents market segments that were not sampled, the amount remaining in products, and an amount that is potentially destroyed.

In market segments with sampling results for more than one process, the representative PPFs selected were generally the highest value for the environmental media and the lowest value for $PPF_{destroyed}$. The amount destroyed is a conservatively low estimate, because the lowest value was generally chosen to represent that market segment. Together, environmental media and destruction account for 88% of the input APFO. What happens to the remaining 12% is undetermined because there are no data that clearly identify the compartments where it is likely to be found.

F. Major Conclusions of the Study

The study objectives were achieved. Detailed information on what happens to APFO when AFD are processed was obtained for 15 individual processes through the survey and samples collected. In an earlier report to EPA, the FMG reported that approximately 15% of the APFO used to make fluoropolymers remained in aqueous dispersions sold to others. The study results enable conclusions to be drawn about what happens to that 15%, as shown in Table 7.

Results are shown in two ways. The first column shows the fraction of the total APFO used to make fluoropolymers, totaling 15%. The second shows the fraction of APFO in AFD in each output stream, totaling to the 100% of the APFO in AFD.

Table 8: Material Balance for APFO Remaining in Aqueous Fluoropolymer Dispersions			
APFO Destinations	Percent of APFO in Fluoropolymer Industry	Percent of Input APFO in AFD	PPF Range
Destroyed in AFD Processing	9%	62%	0 – 81%
Air Streams	2%	16%	0 – 44%
Wastewater Streams	<1%	5%	0 – 22%
Solid Waste Streams	<1%	5%	0 – 18%
Undetermined	1%	12%	Not applicable
Total APFO in AFD	15%	100%	NA

The data in the table must be interpreted with care. The specific PPFs for an individual process can be summed, which must by definition total 100%. For example, if a large amount of APFO was found in the exhaust air stream for a process, there must be a correspondingly lower value in the other categories (solid waste, wastewater, destruction, or remaining in the product) in order to compensate for the greater amount in the exhaust air stream.

1. *Predicted APFO in Waste Streams*

Air streams often are released to the atmosphere during heating steps in a process, but some are treated on site using methods that remove APFO from the air stream, including thermal destruction, wet scrubbing, and particulate filtration. Emission controls on existing air streams are used for purposes of controlling other air pollutants, and may incidentally result in control of APFO. For example, in processes that run at ambient temperature, only small amounts of APFO are released from liquid processes such as formulating coatings and those are in solid form. Typically, a particulate air filter on a mixing tank dust control system will capture APFO along with the dust from the solid materials that are added to a batch. Similarly, in a spray coating operation, most of the APFO detected in the waste streams was in the solid waste coming from the spray booth filters. Processors that use thermal oxidizers or other high temperature control devices have specific air pollution control permit requirements driven by other pollutants.

APFO measured in air sources, including those downstream of existing controls in the small number of facilities that had them, represents 16% of the input APFO in AFD, equal to approximately 2% of the total APFO used in fluoropolymer manufacturing. Much less than 1% of the APFO in AFD is found in air at ambient processing temperatures (up to 65 deg C, 12_deg F). This is consistent with the fact that APFO is a solid at ambient temperature and is a salt dissolved in the water medium. Without a mechanism to disperse the APFO into an air stream, it remains in the liquid.

The amount of APFO found in air waste streams varies at processing temperatures in the range of 300 - 400 deg C (150-204 deg F). Although a large proportion of the AFD is processed in recirculating ovens, less than 11% of the input APFO is found in exhaust air from ovens that operate in this range due to thermal destruction.

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In contrast, a much smaller proportion of the AFD is processed in non-recirculating ovens. Thus, less than 3% of the input APFO in AFD is found in exhaust air from radiant heat ovens that operate in the range of 300 - 400 deg C (572 -752 deg F) and that do not recirculate the process air. Some metal coating processes had intermediate process steps that operated between 150 deg C (302 deg F) and 300 deg C (572 deg F), but all had final processing temperatures in excess of 350 deg C (662 deg F). Valve and pump packing manufacturing was identified as a process that had final processing temperatures between 150 deg C (302 deg F)and 300 deg C (572 deg F).

APFO measured in wastewater sources is less than 1% of the total APFO used in making fluoropolymers. Wastewater sources are collected and treated on site or sent for treatment off site. Some wastewater sources are incinerated and some are treated with other wastewater treatment methods. None of the wastewater from the processes observed in the Study is discharged directly to the environment.

In most of the processes observed, water is used primarily to rinse dispersion containers, process vessels, and processing equipment at ambient temperatures. The volume of cleanup water observed in all but one case was small, less than 0.002 liters per kilogram (0.001 gallons per pound) (dry weight) of AFD processed, and the measured concentrations are low. The PPF_{water} was consistently below 2.8%, except for two processes.

In one process, a wet scrubber is used and process water comes in direct contact with the AFD-treated product. The APFO in that process produced a PPF of 22%. The other process discharges unused AFD bath to its wastewater treatment plant, leading to a wastewater PPF of 6% for this process. However, the total amount of AFD used in these types of processes is less than 16% of the total APFO in AFD. Thus, even taking this into account, the wastewater stream represents 5% of the input APFO from AFD.

APFO measured in solid wastes represents 5% of the input APFO in AFD. Some solid wastes are incinerated, but most are sent to controlled landfills. The solid waste streams that contain the majority of APFO in the solid waste stream are subject only to ambient temperatures; in those wastes that are subject to typical heat treatment temperatures, the measured amounts of APFO were less than 150 ppm (w/w).

2. *Predicted APFO Remaining in Sintered Articles*

Given the mechanisms of APFO destruction, the APFO remaining in articles can be divided between those that are sintered, or subject to high processing temperatures, and those where the AFD are applied and lower drying or baking temperatures (unsintered) are used in the process. The APFO remaining in sintered articles can be estimated by comparing the amounts found in waste that was created after the sintering step. The samples of trim waste from heat-treated product measured in the Study were all less than 150 ppm (w/w), some results were even less than 1 ppm (w/w). Samples of heat-treated product trim waste were collected for glass cloth and film coating processes. The APFO content is expressed on the basis of total coated material, including the substrate. Samples of heat-treated material from a metal coating process were not collected, however, the FDA reported 0.004 to 0.075 ppm.[25]

3. *Predicted APFO Remaining in Unsintered Articles*

APFO remaining in unsintered articles was not measured, as the Study design did not include sampling and analysis of products or articles produced in AFD processing. The APFO content of articles is less than the APFO content of the AFD because the articles are likely to contain other components that do not contain APFO. The typical amount of APFO in AFD is 0.28% by weight.

The DPMB study did not include measurements of the APFO remaining in unsintered articles. The APFO remaining in unsintered products depends somewhat on the processing temperature and the quantities of environmental media generated. Some of the input APFO may transfer to air at temperatures above 150 deg C (302 deg F), but at temperatures below 100 deg C, no mechanisms for transfer to air were found other than spraying the AFD-containing product into the air.

Similarly, there are no mechanisms for destruction of APFO at lower temperatures. Some of the input APFO may transfer to wastewater and solid waste streams depending on the type of process and the quantities generated. While there are always some losses due to sources of solid waste as well as inherent inefficiencies in transferring products between containers, it is likely that a relatively small fraction of the APFO from AFD in such processes will end up in air, wastewater and solid waste streams.

No data were developed on the levels of APFO in air in processes where air temperatures between 100 deg C and 250 deg C were described. However, these processes use a small fraction of the AFD in the U.S. and are not likely to have large quantities in their process streams. The magnitude of the PPF for wastewater or solid waste streams from such processes will likely depend, in part, on the use of air pollution control technology such as baghouses or scrubbers. No estimates of the PPFs for such processes are available.

Absent data on these losses, there are presently no clear data to indicate the APFO remaining in unsintered articles. However, the amounts of AFD used in making these articles represent a small fraction of the total AFD manufactured. The actual APFO content of articles will be evaluated in another study being conducted by the FMG.

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[25] Begley, T.D., page 10.

4. *Estimated APFO Destroyed*

While it was not possible to directly measure the amount of APFO destroyed in processing, destruction is likely to occur in some processes. Given the temperatures and times at which the products are processed and the thermal behavior of APFO reported in the literature, however, it is apparent that a large fraction of any APFO in the products as they enter the final sintering cycle is likely destroyed. Based on the data collected, it is estimated that high-temperature processes will destroy 46 to 90% of the APFO input to the manufacturing process. If the time-temperature profile of the product is above 400 deg C (752 deg F), the amount of residual APFO in the product will be very small. This is consistent with the analysis of solid waste from heat-treated products and publicly available data on APFO extracted from finished articles. The overall level of destruction can be calculated using the material balance results for each process category and the quantity of AFD sold to each process category. The overall destruction level is calculated to be 62% APFO in AFD, or about 9% APFO used in fluoropolymer manufacturing.

IX. Summary

Several key lessons and major conclusions have been drawn from the surveys, observations and sampling. As noted, a large amount of the APFO in AFD is destroyed in the processes. The overall sampling results are amenable to aggregating across multiple process sectors. The APFO detected in wastewater sources is low and can be aggregated across all processes, except for those few market segments that had bath disposal to wastewater and water that had direct contact with AFD. Solid waste emissions are generally small, except for spray-coating processes. While ambient temperature air results are comparable, oven results differ. Ovens with recirculating air are remarkably more effective in destroying APFO than radiant heat ovens and those without recirculation.

The study included a broad and representative sampling of the industry as defined by several different criteria: number of processes, process types, process sectors in the dispersion processing industry, amount of dispersion in use by the study participants, and environmental performance. The breadth and depth of the study provided numerous opportunities to aggregate data across multiple industry sectors and across the air, wastewater, and solid waste media that were sampled. In some cases, certain process parameters were unique, and the air streams from those sources were differentiated from the balance of the industry processes. No processor added APFO to dispersions above that as received from the fluoropolymer manufacturers. Smaller amounts of the APFO were measured in the air, wastewater, and solid waste streams from the industry. Wastewater and solid waste streams are further treated or sent to controlled facilities for disposal. This study demonstrated significant destruction of the input APFO contained in the AFD used in the dispersion processing industry, and support the conclusion that small amounts of input APFO remain in finished products.

Appendix I
Study Protocol

Appendix II
QAPP

Appendix III
Acronym List and Glossary

ABS	Acrylonitrile-Butadiene-Styrene Resin
AFD	aqueous fluoropolymer dispersion
AP	AFD Percentage
APFO	ammonium perfluorooctanoate
Barr	Barr Engineering
C8	APFO, or ammonium perfluorooctanoate
CBI	Confidential Business Information
DPMB	Dispersion Processors Material Balance
DQO	Data Quality Objectives
ECA	Enforceable Consent Agreement
EPA	United States Environmental Protection Agency
EPCRA	Emergency Planning and Community Right-To-Know Act
Exygen	Exygen Research Inc.
FEP	Fluorinated ethylene propylene
FMG	Fluoropolymers Manufacturers Group
FPG	Fluoropolymers Processors Group
HVLP	High Volume Low Pressure (Spray Guns)
IP	Industry Percentage
K&H	Keller and Heckman LLP
KHA	KHA Consulting LLC

LC/MS/MS	High Performance Liquid Chromatography Tandem Mass Spectrometry
NA	Not Applicable
negl	Negligible
PFA	Perfluoroalkoxy polymers
PFOA	Perfluorooctanoic acid
PPF	PFOA Partition Factors defined as the fraction (percent) of the APFO entering the process that leaves the process through various environmental media
PTFE	Polytetrafluoroethylene
PVDF	Polyvinylidene fluoride
QA/QC	Quality Assurance and Quality Control
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RCRAInfo	Resource Conservation and Recovery Act Information Database
SARA	Superfund Amendments and Reauthorization Act
Sintering	Heating at temperatures in excess of the melting point of the fluoropolymer to cause the polymer particles to melt and fuse together, to develop final properties of the polymer.
SPI	The Society of the Plastics Industry, Inc.
TRI	Toxic Release Inventory
TRIS	Toxic Release Inventory System
TSCA	Toxic Substances Control Act

Appendix IV

Survey