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OPPT-2003-0071-0066

Rich Leukroth
01/23/04 10:23 AM

To: dominiak.mary@epa.gov
cc:
Subject: Draft incineration ECA documents 1 of 2 e-mails

This e-mail transmits DRAFT documents for the PFOA incineration ECA activities that will be discussed during the January 27-29 PFOA meetings. Attached are separate files for the fluoropolymer and telomer draft ECA documents. A third file contains Appendices that are common to both ECAs. These draft documents are provided to the Interested Parties as FYI. The Incineration Drafting Committees are not looking for detailed comments from you at this time. A formal opportunity for Interested Party comment will be provided on the DRAFT FINAL document when they become available. These drafts are being distributed: 1) to show the significant progress that has been made in developing the appendices and ECA agreement language since the last PFOA Plenary, and 2) for use during progress report discussions at the January PFOA meetings.

These draft documents incorporate changes as discussed during Drafting Committee teleconference held on 1/21/04 and some but not all changes from the 1/22/04 teleconference. The following identifier highlights those few remaining places where the Drafting Committee continues discussions and/or where additional work remains:

> [NOTE: Drafting Committee discussions are continuing to finalize this section]

Please be familiar with these materials so you can follow along during the discussions next week.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DRAFT #7 TO INTERESTED PARTIES - PFOA ECA PROCESS

ENFORCEABLE CONSENT AGREEMENT FOR THE LABORATORY-SCALE INCINERATION TESTING OF FLUOROPOLYMERS

Docket No. OPPT - 2003 - 0071

[DRAFT 1/27-29/04]
[Month Year]

NOTE TO INTERESTED PARTIES:

This *DRAFT* document is provided to the Interested Parties as FYI. The Drafting Committee is not looking for detailed comments from you at this time. A formal opportunity for Interested Party comment will be provided on the DRAFT FINAL document when it becomes available. This draft is being distributed: 1) to show the significant progress that has been made in developing the appendices and ECA agreement language since the last PFOA Plenary, and 2) for use during progress report discussions at the January PFOA meetings. This draft incorporates changes as discussed during Drafting Committee teleconferences held on 1/21 and 1/22/04. The following identifier highlights those few remaining places where the Drafting Committee continues discussions and/or where additional work remains:

[NOTE: Drafting Committee discussions are continuing to finalize this section]

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ENFORCEABLE CONSENT AGREEMENT FOR THE LABORATORY SCALE INCINERATION TESTING OF FLUOROPOLYMERS

Docket No. OPPT-2003-0071

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I. INTRODUCTION

Under the authority of section 4 of the Toxic Substances Control Act (TSCA), 15 U.S.C. 2603, and 40 CFR Part 790 of the Agency's implementing regulations, the United States Environmental Protection Agency (EPA) and Asahi Glass Fluoropolymers USA, Inc., Daikin America, Inc., Dyneon, LLC, and E.I. du Pont de Nemours and Company (hereinafter collectively "the Companies") enter into this enforceable consent agreement (ECA). This ECA will take effect on the date of publication of the notice in the Federal Register announcing the issuance of the testing consent order (Order) that incorporates this ECA.

On April 16, 2003, EPA initiated a public process to negotiate enforceable consent agreements (ECAs) concerning perfluorooctanoic acid (PFOA) and fluorinated telomers to develop environmental fate and transport information, as well as relevant information to enhance understanding of the sources of PFOA in the environment and the pathways by which human exposure to PFOA is occurring (68 FR 18626; April 16, 2003). The goal of the ECAs resulting from these public discussions is to develop data relevant to identifying the pathway or pathways that result in exposures to PFOA by air, water, soil, or food; and to characterize how PFOA gets into those pathways (including the products or processes that are responsible for the presence of PFOA in the environment). EPA anticipates that the data to be developed under such ECAs will be beyond or supplemental to that of ongoing testing efforts described under industry letters of intent (LOIs) (Refs 1-4). **[OPPT-2003-0012-0007,0012,0013,0016]**

In preparation for the June 6, 2003, public meeting, EPA developed a preliminary framework document outlining data needs that the Agency deemed appropriate to address the outstanding PFOA source and exposure questions identified in the *Federal Register* notice of April 16, 2003 (Ref 5)**[OPPT-2003-0012-0056]**. The intent of EPA's preliminary framework document was to serve as a discussion guide for the June 6, 2003, public meeting and to aid in distinguishing between outstanding EPA data needs and industry LOI commitments. The preliminary framework document was not a predetermined list of information needs defining the outcome of the ECA process.

This ECA provides for a laboratory-scale incineration testing program of fluoropolymers, which is one of the data needs identified in EPA's preliminary framework document for PFOA. On June 6, 2003, the PFOA Plenary Group (consisting of EPA and all interested parties) acknowledged such a testing program as an opportunity for ECA development and tasked the Fluoropolymer Technical Workgroup to work out the details that could be incorporated into an ECA between test sponsors and EPA. On July 9, 2003, the Fluoropolymer Technical Workgroup received proposals from the Companies and EPA for incineration testing of fluoropolymers. Details of this testing program were developed by members of the Fluoropolymer Incineration Subgroup of the Fluoropolymer Technical Workgroup during subsequent meetings. On **[Month/Date]**, 2003, the Fluoropolymer Technical Workgroup acknowledged that this testing program had sufficient merit for consideration by the Plenary Group. On **[Month/Date]**, 2003, the Plenary Group discussed the merit of this testing program and recommended that EPA consider entering into an ECA with test sponsors. The official record for the development of this ECA, including the public version, is established under EPA docket control number [OPPT-2003-0012]. The procedures for ECA negotiations are described at 40 CFR 790.22(b). The official record for the testing conducted under this ECA is Docket No. OPPT-2003-0071

II. TEST SUBSTANCES

For the purposes of testing under this ECA the chemicals listed in Appendix A.1[1] will be combined to form four composites (see Appendix A.3 and A.4). These four composites are considered the subject test substances under this ECA These composites are representative of fluoropolymer products manufactured by the Companies that are currently available in the marketplace. The Companies will provide the fluoropolymers specified in Appendix A.1 for incorporation into the composites that will be tested under this ECA.[2] Criteria for the selection of each composite to be tested under this ECA are described in Appendix A.2 of this ECA[1]. The composition of each composite is described in Appendix A.3 of this ECA [1]. The four composites to be tested are defined for purposes of this ECA as:

(A) Dry Non-Melt PTFE Resin Composite: Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0,

(B) Dry Melt Fluoropolymer Resin Composite: (containing: 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; 1-Hexene, 3,3,4,4,5,5,6,6,6-nonafluoro-, polymer with ethene and tetrafluoroethene, CAS No. 68258-85-5; and, 1-Propene,

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[1] There is a Public and CBI version of Appendices A.1, and A.3 because some of the Companies have asserted that details describing one or more of the chemicals subject to this ECA are entitled to treatment as TSCA confidential business information (CBI) (see Part XV of this ECA regarding confidentiality of information).

[2] See the Tables in Part XXIV. of this ECA for the chemicals to be supplied by each Company.

1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene, CAS No. 35560-16-8),

(C) Dry Non-Melt Fluoroelastomer Gum Composite: (containing: 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene, CAS No. 9011-17-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; 1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 54675-89-7; 1-Propene, polymer with tetrafluoroethene, CAS No. 27029-05-6; Ethene, tetrafluoro-, polymer with trifluoro(trifluoromethoxy) ethene, CAS No. 26425-79-6; and, Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene, CAS No. 9010-75-7; and ??generic name??, **Accession No. ??????**, and

(D) Aqueous Fluoropolymer Dispersions Composite: (containing: Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy) ethene, CAS No. 31784-04-0; Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; and 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0.

The procedure for constructing each composite is described in Appendix A.4 to this ECA[1]. The polymer components for each composite will be unfilled first quality product polymer, substantially free of inorganic constituents. Each component of the four composites to be tested under this ECA will be accompanied by a certificate of analysis showing it to meet applicable product specifications.

III. OBLIGATION OF SIGNATORY COMPANIES

A. The Companies are bound by the terms of this ECA as specified below.

B. Each Company shall be responsible for supplying the test substance(s) it manufacturers for incorporation into the composite(s) to be tested under this ECA, as specified on each Company signature page and in Appendix A.3. The schedule for the testing program includes the deadline date by which the Companies must submit their contribution(s) to the facility(ies) that will be assembling the composites to be tested under this ECA. Any Company failing to comply with this ECA requirement will be in violation of this ECA as described in 40 CFR 790.65 (see Part XII of this ECA). In the event that one or more of the Companies are in violation as described above then the remaining Companies will inform EPA of the problem and request an EPA determination on how to proceed with the testing program described under this ECA. Each Company required to contribute to a particular composite is obligated to complete the testing required by this ECA for that composite. A Company shall not be responsible for any failure to perform its obligation under this ECA that is caused by circumstances beyond its control, that the Company could not have prevented through the exercise of due diligence. Under such circumstances the Company will consult with EPA to reach agreement on what modifications, if any, are needed in the test plan or scope of testing (see Part X of this ECA regarding modification to this ECA as contained in 40 CFR 790.68).

C. The Companies recognize that to implement this ECA, EPA will issue an Order under section 4 of TSCA that incorporates the terms of this ECA (see Appendix F). The Companies agree that all terms of this ECA will take effect on the date of publication of the notice in the Federal Register announcing the issuance of the Order that incorporates this ECA, and all time periods that begin on the effective date, will be treated as beginning on that publication date.

IV. PRINCIPAL TEST SPONSOR

The Companies have identified the Fluoropolymer Manufacturers Group (FMG), to communicate with EPA about schedules, study plans, protocols, test standards, and other aspects of the testing program. EPA and the Companies agree that FMG has no legal responsibility for complying with this ECA. Responsibility for complying with the ECA rests at all times with the Companies.

V. PURPOSE OF THE TESTING PROGRAM

The purpose of the testing program specified by this ECA is to assess the potential for waste incineration of fluoropolymers (see Part II and Appendix A.1 of this ECA) to emit PFOA, based on quantitative determination of potential exhaust gas levels of PFOA that may emanate from laboratory-scale combustion testing under conditions representative of typical municipal waste combustor operations in the United States.

EPA believes that these incineration studies of fluoropolymers will develop data needed by the Agency to determine whether municipal and/or medical waste incineration of fluoropolymers is a potential source of PFOA that may contribute as a pathway to environmental and human exposures. The data may also be used to inform screening level human and environmental exposure assessments. In addition, the data may also be used by other Federal agencies (e.g., the Agency for Toxic Substances and Disease Registry (ATSDR), the National Institute for Occupational Safety and Health (NIOSH), the Occupational Safety and Health Administration (OSHA), and the Consumer Product Safety Commission (CPSC), the Food and Drug Administration (FDA)) in assessing chemical risks and in taking appropriate actions within their programs. It is intended that the data generated under this ECA will identify whether the incineration of fluoropolymers contributes to the sources and pathways of environmental and human exposure to PFOA.

VI. SCOPE OF THE PROGRAM

The scope of this testing program is described in Parts VII and VIII below and will consist of the testing listed in Table 1 in accordance with the test standards specified in Table 1 and described in Appendix B.1 and C.1 - C2 as annotated by Appendix D.1- D.4 to this ECA ("Test Standards") and submitting the reports and documents specified in Table 1 in accordance with the deadlines set forth in Table 1 and described in Appendices C.1 - C.2 and E.1- E.2.

VII. DESCRIPTION OF THE TESTING PROGRAM

The program has two segments as follows: Phase I PFOA Transport Testing and Phase II Fluoropolymer Incineration Testing.

A. Phase I PFOA Transport Testing: Phase I will consist of quantitative transport efficiency testing for PFOA. Phase I testing for PFOA transport efficiency is specified in the Phase I PFOA Transport Testing segment of Table 1 and described in Appendix C.1 as annotated by Appendix D.1 and D.2. At the conclusion of Phase I testing, the Companies, will provide EPA with a letter report summarizing the results. In the event that the transport efficiency of PFOA or total fluorine (as determined by the formulas in Appendix C.1) is equal to or greater than 70%, testing will proceed to Phase II Fluoropolymer Incineration Testing. In the event the transport efficiency of PFOA or total fluorine (as determined by the formulas in Appendix C.1) is less than 70%, the Companies will initiate a technical consultation with EPA (see Part VII. B. and Part VIII of this ECA).

B. Phase II Fluoropolymer Incineration Testing: This testing, specified in the Phase II Fluoropolymer Incineration Testing segment of Table 1 and described in Appendix C.2 as annotated by Appendices B.1, D.1, D.2, D.3, D.4, and E.2; and will include the following for each fluoropolymer composite to be tested under this ECA: 1) elemental analysis, 2) combustion stoichiometry, 3) thermogravimetric analysis, 4) laboratory-scale combustion testing, and, 5) if required under this ECA,[3] release assessment reporting.

VIII. PHASE I TECHNICAL CONSULTATION

A. Following completion of Phase I and prior to the initiation of Phase II, the Companies will submit a letter report to EPA with the results for the recovery across the laboratory-scale thermal reactor system, as determined from Phase I testing.

B. If the recovery for either PFOA or Total Fluorine (as determined by the formulas in Appendix C.1) is greater than or equal to 70%, the Companies will proceed to Phase II testing.

C. If the recovery for both PFOA and Total Fluorine (as determined by the formulas in Appendix C.1) is less than 70%, a Technical Consultation will be held between the Companies and EPA. The objective of the Technical Consultation will be to reach agreement on how to proceed. The technical consultation will review the outcomes of the Phase I PFOA Transport Efficiency Testing, discuss the feasibility of proceeding with Phase II Testing as described in this ECA, and discuss whether additional modifications are needed to the test standards and/or protocols described in Appendices B, C and D for Phase I PFOA Transport Testing and/or Phase II Fluoropolymer Incineration Testing. Specifically, the technical consultation will address: (1) whether the data from the Phase I PFOA Transport Testing segment provide a sufficient basis for conducting the laboratory-scale incineration testing specified in the Phase II Fluoropolymer Incineration Testing segment; (2) the nature and scope of any additional Phase I work that may be required prior to the commencement of Phase II Testing and reporting (e.g., modifications to the Advanced Thermal Reactor System) as described in Part VII. B. of this ECA), and/or (3) the nature and scope of modifications to the protocols and test standards for Phase I and/or Phase II testing, or the identification of additional testing, that may be needed to complete the testing under this ECA.

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[3] In the event that Phase II Fluoropolymer Incineration Testing identifies measurable levels of PFOA (where measurable PFOA is defined to be at or above the limit of quantitation (LOQ) as defined in Appendix D.2) resulting from the incineration testing for any or all of the fluoropolymer composites tested under this ECA (see Part II and Appendix A.1 - A.4 to this ECA), the Companies will prepare a release assessment report (see Table 1 and Appendix E.2 to this ECA) to place in perspective the relevance of such measurable levels in the laboratory-scale incineration testing results with respect to full-scale municipal and/or medical waste incinerator operations in the United States.

Possible outcomes of the Technical Consultation include, the following:

1. An agreement to conduct additional Phase I testing, and the schedule and standards for such testing, to inform whether and under what conditions to conduct Phase II testing.

2. An agreement to proceed into Phase II testing with or without agreed-to modifications to plans, test standards and schedules for Phase II testing.

3. An agreement to conduct such other testing, and the schedule and standards for such testing, in Phase II that the Companies and EPA agree may be appropriate, in light of Phase I results, to assist in determining the potential for release of PFOA from fluoropolymers during waste incineration.

4. No agreement on a path forward, in which case the Companies' obligations to conduct testing or reporting beyond Phase I PFOA Transport Testing as described in this ECA are terminated.

D. EPA shall place in the docket (OPPT-2003-0071) a summary of any Technical Consultation that is held under this paragraph. In the event modifications to the testing program are agreed to, EPA and the Companies will revise this ECA, as well as Table 1 and the Appendices, as appropriate.

IX. STANDARDS FOR CONDUCTING TESTING

A. Testing for the laboratory-scale incineration of the fluoropolymer test substance composites described in Part II of this ECA which contain the fluoropolymers listed in Appendix A.1 of this ECA must be conducted in accordance with the Test Standards listed in Table 1 and described in Appendices B.1 and C.1 - C.2 as annotated in Appendices D.1- D.3 to this ECA. Certain provisions of these Test Standards are considered to be mandatory and are referred to as "requirements." These requirements are identified by the use of the word "shall" in the text of the Test Standard. For the purpose of this ECA, the words "will" and "must," if they appear in the Test Standards, are considered equivalent to the word "shall" and therefore delineate a test requirement to be followed or met.

Provisions that are not mandatory, and are therefore only recommended, are identified by the use of "should" statements. In the event such "should" provisions are not followed, the Companies will not be deemed by EPA to be in violation of this ECA and will not be subject to penalties or other enforcement actions, as described in Part XII. of this ECA. However, in such cases, EPA will use its professional judgement to determine the scientific adequacy of the test results and any repeat testing that is determined by EPA to be necessary will be required either under a separate ECA or pursuant to a rule promulgated under section 4(a) of TSCA, 15 U.S.C. 2603(a).

B. The Companies and EPA will consult in a good faith effort to consider the need for Test Standard modifications if either EPA or the Companies desire such modifications. Modifications to this ECA will be governed by 40 CFR 790.68 (see Part XI. of this ECA).

***** FOLLOW-ON DISCUSSION POINT FOR DRAFTING COMMITTEE:**
[SUMMARY: Discussions are continuing on: (a) appropriate tailoring of GLP requirements to meet the special needs of this test program, and (b) a table providing guidance for the development of quality assurance project plans.]

{original text under discussion}

C. All testing required by this ECA must be conducted in accordance with the EPA Good Laboratory Practice Standards (GLPS) found at 40 CFR part 792.

X. STUDY PLAN(S) AND QUALITY ASSURANCE PROJECT PLAN(S) (QAPP)

The Companies will submit a study plan to EPA for each test conducted pursuant to this ECA prior to the initiation of testing in accordance with 40 CFR 790.62. (For this ECA, EPA will not require the plan(s) under this Part of the ECA to be submitted "no later than 45 days prior to the initiation of testing," as specified at 40 CFR 790.62(a)). The content of the study plan(s) submitted to EPA will comply with 40 CFR 790.62(b). This ECA and/or its appendices satisfy the applicable requirements of 40 CFR 790.62(b)(2), (8), (9), and (10). A study plan may cross reference the applicable provisions of the ECA and/or its appendices to satisfy these requirements. Also ~~pursuant to Part IX. (C) of~~ for this ECA, the Companies must submit Quality Assurance Project Plan(s) (QAPP) prepared in accordance with EPA guidance.[4] Modifications

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[4] Guidance for developing Quality Assurance Project Plans can be found in the EPA document EPA QA/R-5: *EPA Requirements for Quality Assurance Project Plans,* prepared by: Office of Environmental Information, EPA, March 2001. This is also available from the EPA website at (continued...)

to the study plan(s) under this part of the ECA will be governed by the procedures of 40 CFR 790.62(c) except that the 15 day time periods in 40 CFR 790.62(c) (2) and (3) will be 45 day time periods. All study plan(s) will become part of the official record (Docket Control Number [OPPT-2003-0071).

XI. MODIFICATIONS TO THIS ENFORCEABLE CONSENT AGREEMENT

Modifications to this ECA, if any, will be made according to the procedures contained in 40 CFR 790.68.

XII. FAILURE TO COMPLY WITH THE ENFORCEABLE CONSENT AGREEMENT

The Companies acknowledge that a violation of the requirements of this ECA will constitute a "prohibited act" under section 15(1) of TSCA, 15 U.S.C. 2614(1), and will trigger all provisions applicable to a section 15 violation. Further information regarding the implications of failure to comply with the consent agreement is provided in 40 CFR 790.65.

XIII. EPA MONITORING OF ENFORCEABLE CONSENT AGREEMENT TESTING

EPA may conduct monitoring activities of the testing conducted under this ECA such as laboratory inspections and study audits, as permitted under section 11 of TSCA, 15 U.S.C. 2610.

XIV. SUBMISSIONS TO EPA AND CONFIDENTIALITY OF INFORMATION

A. All reporting required by this ECA must be submitted by the Companies to EPA by the dates specified in Table 1 unless otherwise authorized by EPA pursuant to 40 CFR 790.68. A paper copy of a document shall be deemed submitted when it is either postmarked or placed in the hands of a commercial courier service for overnight delivery to EPA at the appropriate address specified in Part XIV. B. of this ECA. Hand-delivered documents are deemed submitted upon receipt at the appropriate address specified in Part XIV. B. of this ECA. Electronically transmitted documents are deemed delivered upon transmission and must follow the procedures for electronic submissions specified in Part XIV.B. of this ECA. Under any of the above

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[4] (...continued)
http://epa.GOV/Quality/qs-docs.

circumstances, it is the responsibility of the Companies to maintain appropriate documentation for proof of transmittal for all reporting required by this ECA.

In accordance with 40 CFR 790.62 (d), the Companies will submit interim progress reports to EPA informing the Agency of any proposed changes in standards for the development of data, study plans, or test schedules, and communicating with the Agency about laboratory inspections and other matters affecting the progress of testing. The schedule for interim progress reports is specified in Table 1 of this ECA. The information required in interim progress reports is specified in Appendix E.1.

B. All documents submitted to EPA under this ECA must be identified by the Docket ID Number (OPPT-2003-0071) and the name: ECA on Laboratory-Scale Incineration Testing of Fluoropolymers.

Submissions made by mail should be sent to: Document Control Office (7407M), Office of Pollution Prevention and Toxics (OPPT), Environmental Protection Agency, 1200 Pennsylvania Avenue, NW, Washington, DC 20460-0001.

Submissions made by hand delivery or courier should be delivered to: OPPT Document Control Office (DCO) in the EPA East Building, Room 6428, 1201 Constitution Avenue, NW, Washington, DC and marked Attention: Docket ID Number OPPT- 2003 -0071. The DCO is open from 8 a.m. to 4 p.m., Monday through Friday, excluding legal holidays. The telephone number for the DCO is (202) 564-8930.

Submissions made electronically should be sent to: OPPT Document Control Office at http:www.oppt.ncic@epa.gov, Attention: Docket OPPT-2003-0071. Electronic submissions do not supersede the requirements of Part XIV. C. of this ECA. Electronic submissions for all reporting required by this ECA must be submitted as attachments to the e-mail and must be in text-searchable, PDF format. The e-mail transmitting any report required by this ECA and all electronic attachments will be included as part of the submission. E-mail addresses are automatically captured by the EPA e-mail system and become part of the submission that is placed in the official public docket, and will be made available in the EPA electronic public docket. Upon receipt of the electronic submission, a "receipt date" is entered into the metadata to signify the date the document(s) submitted by the Company(ies) was received by EPA. EPA is not responsible for failure to meet a date of submission requirement if the EPA fire wall rejects an electronic submission containing a virus or other adverse electronic coding. It is the obligation of the submitter to confirm that: 1) electronic submissions are received by EPA on the date of transmission, 2) the electronic submission and all attachments are legible, and 3) the electronic submission and all attachments meet the electronic format requirements of the EPA Document Control Office. Do not submit any report containing confidential business information (CBI) to EPA by e-mail. For submissions containing CBI see Part XIV.D of this ECA.

C. The Companies must submit six (6) paper copies of each version (Public and CBI) for all reports described in Table 1 and Part VII A. and B. of this ECA. In addition, an electronic file, on a disk or CD ROM, of all documents submitted under this ECA (marked as CBI where appropriate and in text-searchable, PDF format) will be provided to EPA. To avoid damage caused by mail scanning technologies, the electronic file on disk or CD ROM must be hand delivered or sent by courier to the address cited in Part XIV. A.. See Part XIV. D. regarding submissions containing CBI.

D. Any document submitted to EPA that contains data or information for which a Signatory Company makes a claim of confidentiality (see Part XV of this ECA), must be submitted as two separate versions. One version must be complete, with the information being claimed as confidential marked in the manner described under 40 CFR 790.7. The other, public version must be identical in all respects except that all of the information claimed as confidential shall be redacted. EPA will place the public version in the Agency's docket. The complete version will be treated in accordance with EPA confidentiality regulations in 40 CFR part 2 and 40 CFR 790.7.

Data or other information that are considered to be CBI must not be submitted electronically to EPA by e-mail. Any part or all of data or other information claimed as CBI must be so marked. If the CBI submission is on diskette or CD ROM, mark the outside of the diskette or CD ROM as CBI and then identify electronically within the diskette or CD ROM the specific information that is CBI. Information marked as CBI will not be disclosed except in accordance with procedures set forth in 40 CFR part 2 (see Part XV of this ECA).

Any claims of confidentiality for information submitted under this ECA will be made under the terms of 40 CFR 790.7. If no claim of confidentiality is made by the submitter of the information at the time of submission, the information will be deemed by EPA, in accordance with 40 CFR 790.7, to be public, and may be made available to the public without further notice to the submitter. Information claimed as confidential will be treated in accordance with the procedures in 40 CFR part 2 established pursuant to section 14 of TSCA, 15 U.S.C. 2613.

XV. PUBLICATION AND DISCLOSURE OF TEST RESULTS

All results of testing conducted pursuant to this ECA will be announced to the public by EPA in accordance with the procedures specified in section 4(d) of TSCA, 15 U.S.C. 2603(d). Disclosure by EPA of data generated by such testing to the public or other government agencies will be governed by section 14(b) of TSCA, 15 U.S.C. 2613(b), and 40 CFR part 2. The CBI

version of a document will only be provided to another U.S. government organization in compliance with the procedures described in the OPPTS TSCA CBI Procedure Manual.
[NOTE: Drafting Committee discussions are continuing to finalize this section]

XVI. OTHER RESPONSIBILITIES OF THE COMPANIES

A. The Companies will comply with the notification requirements of section 12(b)(1) of TSCA, 15 U.S.C. 2611(b)(1), and 40 CFR part 707, subpart D, if they export or intend to export ethene, tetrafluoro-homopolymer (PTFE) (CAS No. 9002-84-0) or any of the composite test substances described in Part II and Appendix A.3 of this ECA. Any other person who exports or intends to export ethene, tetrafluoro-homopolymer (PTFE) (CAS No. 9002-84-0) or any of the composite test substances described in Part II and Appendix A.3 of this ECA is subject to the above cited export notification requirements

B. If any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA become subject to a rule promulgated under TSCA section 5(a)(2), 15 U.S.C. 2604(a)(2), governing significant new uses of any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA, then the Companies will be subject to the data submission requirements imposed by section 5(b)(1)(A) of TSCA, 15 U.S.C. 2604(b)(1)(A), as if the testing under this ECA had been required by a TSCA section 4 test rule.

XVII. SEVERABILITY OF ENFORCEABLE CONSENT AGREEMENT PROVISIONS

In the event that one or more provisions of this ECA are determined by a court decision to be unenforceable, the remaining provisions of this ECA will not be presumed to be valid, and EPA will either initiate a rulemaking proceeding to require testing or publish in the Federal Register the reasons for not initiating such a proceeding.

XVIII. FINAL AGENCY ACTION

For purposes of 5 U.S.C. 704, publication of the FR notice announcing the issuance of the Order incorporating this ECA constitutes final agency action..

XIX. PUBLIC RECORD

EPA has established a public record which will contain this ECA, the Order that incorporates this ECA, the Federal Register notice announcing issuance of the Order incorporating this ECA, and any and all relevant information, subject to the confidentiality provisions of section 14(b) of TSCA and 40 CFR part 2. The official record for this ECA, including the public version, which does not include any information claimed as CBI, has been established under Docket Control Number OPPT-2003-0071.

An electronic version of the public docket is available through EPA's electronic public docket system, EPA Dockets. EPA Dockets may be accessed at http://www.epa.gov/edocket/ to access the index listing of the contents of the official public docket, and to access those documents in the public docket that are available electronically. Although not all docket materials may be available electronically, (for example the materials in the original dockets for this action, [AR-226 and OPPTS-2003-0012], or materials under copyright), can be access any of the publicly available docket materials through the EPA Docket Center, Rm. B102-Reading Room, EPA West, 1301 Constitution Ave., NW., Washington, DC. For materials available in the electronic docket, once in the system, select "search," then key in the appropriate Docket ID number (OPPT-2003-0071).

XX. EFFECTIVENESS

This ECA may be signed in separate counterparts. This ECA will not be effective unless signed by each of the Companies and by EPA. This ECA will take effect on the date of publication of the Federal Register notice announcing the issuance of the Order that incorporates this ECA.

XXI. RIGHTS OF THE COMPANIES

By signing this ECA, the Companies waive their right to challenge EPA's authority to assess penalties for violations of the terms of this ECA. This waiver does not affect any other rights that the Companies may have under TSCA, including the right to dispute the amount of any penalty or to dispute factually whether a violation of the terms of this ECA has occurred, or to seek judicial review of any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA.

XXII. RESERVATION OF RIGHTS BY COMPANIES

By signing this ECA, the Companies are not admitting that the requirements of TSCA Section 4 have been satisfied for promulgating a test rule to generate the data required by this ECA.

The Companies contend that the documents generated for the incineration testing program under this ECA are protected from public disclosure under 5 U.S.C. section 552(b)(4) and 15 U.S.C. section 2613(a) and do not constitute studies subject to disclosure under 15 U.S.C. section 2613(b). Accordingly, the public information disclosure provisions of this ECA are, in the view of the Companies, a waiver of legal rights.

XXIII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR

The Principal Test Sponsor is:

Fluoropolymer Manufacturers Group
[? Name of technical contact person ?]
[? ADDRESS ?]
[? Phone Number ?]

The Companies subject to this ECA are:

Asahi Glass Fluoropolymers USA, Inc.
[? ADDRESS ?]

Daikin America, Inc.
[? ADDRESS ?]

Dyneon, LLC
[? ADDRESS ?]

E.I. du Pont de Nemours and Company
[? ADDRESS ?]

Special Page Header: ECA Copy # 3 Asahi Glass Fluoropolymers USA, Inc.

XXIV. SIGNATURE

TEST SPONSOR

Asahi Glass Fluoropolymers USA, Inc.[1]

ECA Subject Chemicals for Asahi Glass Fluoropolymers USA, Inc.		
CAS No.	**Chemical Name**	**Composite(s)**

Company technical contact person for handling correspondence marked as "Confidential"

Name: ____________________
Title: ____________________
Address: ____________________
Phone Number: ____________________

Date:______________

____
[? NAME ?]
[? TITLE ? e.g., Senior Vice President]
Asahi Glass Fluoropolymers USA, Inc.
[? ADDRESS ?]

---

[1] Data in the table lists the chemical(s) and composite contributions for which Asahi Glass Fluoropolymers USA, Inc. is responsible. The Company developed these data in response to EPA's letter of January 6, 2004. There may be both a Public and CBI version of this page in those instances where the Company has asserted that data in this table are considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XIV.D. of this ECA regarding confidentiality of information) .

Special Page Header: ECA Copy # 4 Daikin America, Inc.

XXIV. SIGNATURE

TEST SPONSOR

Daikin America, Inc.[1]

ECA Subject Chemicals for Daikin America, Inc.		
CAS No.	**Chemical Name**	**Composite(s)**

Company technical contact person for handling correspondence marked as "Confidential"

Name: ____________________
Title: ____________________
Address: ____________________
Phone Number: ____________________

Date:____________

____
[? NAME ?]
[? TITLE ? e.g., Senior Vice President **]**
Daikin America, Inc.
[? ADDRESS ?]

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[1] Data in the table lists the chemical(s) and composite contributions for which Daikin America, Inc. is responsible. The Company developed these data in response to EPA's letter of January 6, 2004. There may be both a Public and CBI version of this page in those instances where the Company has asserted that data in this table are considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XIV. D. of this ECA regarding confidentiality of information) .

Special Page Header: ECA Copy # 5 Dyneon, LLC

XXIV. SIGNATURE

TEST SPONSOR

Dyneon, LLC[1]

ECA Subject Chemicals for Dyneon, LLC		
CAS No.	**Chemical Name**	**Composite(s)**

Company technical contact person for handling correspondence marked as "Confidential"

Name: ____________________
Title: ____________________
Address: ____________________
Phone Number: ____________________

Date:______________ ________________________

[? **NAME** ?]
[? **TITLE** ? **e.g.,** Senior Vice President]
Dyneon, LLC
[? **ADDRESS** ?]

---

[1] Data in the table lists the chemical(s) and composite contributions for which Dyneon, LLC is responsible. The Company developed these data in response to EPA's letter of January 6, 2004. There may be both a Public and CBI version of this page in those instances where the Company has asserted that data in this table are considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XIV. D. of this ECA regarding confidentiality of information) .

Special Page Header: ECA Copy # 6 E.I. du Pont de Nemours and Company

XXIV. SIGNATURE

TEST SPONSOR

E.I. du Pont de Nemours and Company[1]

ECA Subject Chemicals for E. I. du Pont de Nemours and Company		
CAS No.	**Chemical Name**	**Composite(s)**

Company technical contact person for handling correspondence marked as "Confidential"

Name: ______________________
Title: ______________________
Address: ______________________
Phone Number: ______________________

Date:______________ ______________________________

[? **NAME** ?]
[? **TITLE** ? **e.g.,** Senior Vice President]
E.I. du Pont de Nemours and Company
[? **ADDRESS** ?]

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[1] Data in the table lists the chemical(s) and composite contributions for which E.I. du Pont de Nemours and Company is responsible. The Company developed these data in response to EPA's letter of January 6, 2004. There may be both a Public and CBI version of this page in those instances where the Company has asserted that data in this table are considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XIV. D. of this ECA regarding confidentiality of information) .

Special Page Header:

ECA Copy # 1 EPA PUBLIC VERSION
CONTAINS NO CONFIDENTIAL BUSINESS INFORMATION

Special Page Header:

ECA Copy # 2 EPA CBI VERSION
CONTAINS CONFIDENTIAL BUSINESS INFORMATION

XXIV. SIGNATURE

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Date:____________ ______________________________

Stephen L. Johnson
Assistant Administrator
Office of Prevention, Pesticides, and Toxic Substances

Address:

U.S. Environmental Protection Agency
Office of Prevention, Pesticides, and Toxic Substances
Ariel Rios Building
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460

Table 1 REQUIRED TESTING, TEST STANDARDS, REPORTING AND OTHER REQUIREMENTS FOR THE LABORATORY-SCALE INCINERATION TESTING OF FLUOROPOLYMERS

Phase I PFOA Transport Testing	**Test Standard or ECA Requirement**	**Deadline for Final Report (Months)** [1]
Phase I Study Plan(s)	40 CFR 790.62 (b) as annotated by Part X of the ECA	?TBD ? [3]
Phase I QAPP submission	see Appendix ?? G ?? to the ECA	?TBD ? [3]
Quantitative PFOA transport analysis [2]	See appendix C.1 as annotated in appendix D.?)	?TBD ? [4] [5]

---

[1] Interim progress reports must be submitted by the Companies to EPA every 6 months beginning six months from the effective date of the Order that incorporates this ECA until the end of the ECA testing program (see Part XIV and Appendix E.1 of the ECA).

[2] At the conclusion of Phase I PFOA transport efficiency testing, and prior to initiation of Phase II, the Companies, will provide a letter/report to EPA summarizing the results of Phase I testing (see Part VII. A. of the ECA). In the event that the transport efficiency of PFOA or of total fluorine (as determined by the formulas in Appendix C.1) is greater than or equal to 70% then testing will proceed to Phase II Incineration Testing. In the event that the transport efficiency of PFOA or of total fluorine (as determined by the formulas in Appendix C.1) is less then 70% then the Companies will initiate a Technical Consultation with EPA to determine under what conditions Phase II testing can proceed. The outcomes of the Technical Consultation are described in Part VIII of this ECA.

[3] Number of months after the effective date of the Order that incorporates this ECA when submission is due.

[4] **[NOTE: Drafting Committee discussions are continuing to finalize this section]**

[5] **[NOTE: Drafting Committee discussions are continuing to finalize this section]**

Phase II Fluoropolymer Incineration Testing	Test Standard or ECA Requirement	Deadline for Final Report (Months)[1]
Phase II Study Plan(s)	40 CFR 790.62 (b) as annotated by Part X of the ECA	? TBD ? [3]
Phase II QAPP submission	See Appendix ?? G ?? of the ECA	? TBD ? [3]
Receipt of component chemicals by formulating laboratory(ies) / 3rd Party (?)	See Part XXIV and Appendix A.3 of the ECA	? TBD ? [7]
Elemental analysis [6]	See Appendix C.2.1 of the ECA	? TBD ? [8]
Combustion stoichiometry [6]	See Appendix C.2.2 of the ECA	? TBD ? [8]
Thermogravimetric analysis [6]	ASTM E 1868-02 (as modified by Appendix B.1 of the ECA)	? TBD ? [8]
Laboratory-scale combustion testing [6]	See Appendix C.2.4 of the ECA (as annotated by Appendix D.1, D.2, D.3, and D.4 of the ECA)	? TBD ? [8]
Release assessment report [6]	Ssee Appendix E.2 of the ECA	? TBD ? [8] [9]

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[6] The results of this testing will be provided in the final report for Phase II testing (see Appendix C.2.5 of the ECA).

[7] [NOTE: Drafting Committee discussions are continuing to finalize this section]

[8] [NOTE: Drafting Committee discussions are continuing to finalize this section]

[9] [NOTE: Drafting Committee discussions are continuing to finalize this section]

APPENDIX A

TEST SUBSTANCES

A.1 List of Chemical Components of the Composites
A.2 Rationale for Selecting Composites to be Tested
A.3 Composition of Composites to be Tested
A.4 Preparation of Composites to be Tested

PUBLIC VERSION - CONTAINS NO CBI

APPENDIX A.1

LIST OF CHEMICAL COMPONENTS OF THE COMPOSITES[1]

The following table lists the thirteen commercial fluoropolymer chemicals (made using ammonium perfluorooctanoate (APFO)) that are the subject to this ECA.

The identities of the fluoropolymers (made using APFO) that are components of the composites that are subject to this ECA were provided to EPA as support documentation of the Companies' LOI commitments. Some of this documentation, including certain aspects related to the identity of the test substance as described in Part II of this ECA and the table below, may contain Confidential Business Information (CBI). In such instances EPA creates a comprehensive database for evaluation and comparison, and, when possible, provides a public version sanitized of CBI.

Subsequent analysis of the list of fluoropolymers received by EPA supported the conclusion that the individual chemicals listed below are representative of all known commercial fluoropolymer chemicals and the basic chemistries are represented by the four composite test substances that are subject to testing under this ECA (i.e., dry melt fluoropolymer resin, dry non-melt PTFE homopolymer resin/gum, dry non-melt fluoroelastomer resin/gum, aqueous fluoropolymer dispersions) (see ECA Appendix A.2 and A.3). The fluoropolymer structure is predominantly -(CF2)x- which is a potential source of PFOA. For all fluoropolymer products used in commerce, the -(CF2)- moiety is common to all polymers and the composites to be tested under this ECA testing program (see Appendix A.2-A.4) are representative of the individual component and non-component fluorochemicals.

---

[1] There is a Public and CBI version of Appendix A.1 because the Companies have asserted that details describing their chemical(s) are considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XIV. D. of this ECA regarding confidentiality of information).

FLUOROPOLYMERS SUBJECT TO THIS ECA

No.	CAS No.	Chemical Name
1	9002-84-0	Ethene, tetrafluoro-, homopolymer
2	25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene)
3	26655-00-5	Propane,1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl0oxy]-, polymer with tetrafluoroethene
4	25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene
5	68258-85-5	1-Hexene, 3,3,4,4,5,5,6,6,6,-nonafluoro-, polymer with ethene and tetrafluoroethene
6	35560-16-8	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene
7	9011-17-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene
8	54675-89-7	1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene
9	27029-05-6	1-Propene, polymer with tetrafluoroethene
10	26425-79-6	Ethene, tetrafluoro-, polymer with trifluoro(trifluoroethoxy)ethene
11	9010-75-7	Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene
12	31784-04-0	Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene
13	CBI **Accession No. ????**	**??generic name ??**

APPENDIX A.2

RATIONALE FOR SELECTING COMPOSITES TO BE TESTED

APPENDIX A.2

2. Selection

Review of Figure A-1 demonstrates that fluoropolymers industry products can be divided into 3 broad categories representative classes as follows:

- Dry melt resins
- Dry non-melt resins and gums
- Aqueous dispersions

These three broad categories can in turn be divided into four representative classes as follows:

- Dry melt resins
  1. FEP, PFA, THV, ETFE, HTE

- Dry non-melt resins and gums
  2. PTFE resin
  3. Fluoroelastomer gums

- Aqueous dispersions
  4. PTFE, FEP, PFA, THV

Composite samples of each of these four representative classes were selected as the test substance for this testing program in order to represent the entire range of fluoropolymers involved.

PUBLIC VERSION - CONTAINS NO CBI

APPENDIX A.3

COMPOSITION OF COMPOSITES TO BE TESTED[1]

[NOTE: Drafting Committee discussions are continuing to finalize this section]

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[1] There is a Public and CBI version of Appendix A.3 because the Companies have asserted that details describing their chemical(s) are considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XIV. D. of this ECA regarding confidentiality of information).

DRAFT
10-24-03

APPENDIX A

Appendix A.3

1. Identification

The four composite test substances for this test program will be prepared from the flucropolymers identified in the March 14, 2003 Letter of Intent (LOI) submitted by the Society of the Plastics Industry on behalf of the four LOI signatories (as corrected). The specific fluoropolymer types (with CAS numbers and associated monomers) going into each of the four composite test substances (grouped as shown) are presented in Table A-1 below. Each fluoropolymer used in each relevant test substance composite will have been made using APFO.

Table A-2. Test Substance Composites by Type and CAS Number

Test Substance	Fluoropolymer Type	CAS Number	Associated Monomers
Composite 1 - PTFE resin (dry non-melt)	PTFE	9002-84-0	TFE
Composite 2 - Dry melt resins	FEP	25067-11-2	TFE, HFP
	PFA	26655-00-5 31784-04-0	TFE, PPVE TFE, PEVE
	THV	25190-89-0	TFE, HFP, VDF
	ETFE	68258-85-5	TFE, E
	HTE	35560-16-8	TFE, HFP, E
Composite 3 - Fluoroelastomers (dry non-melt)	Fluoroelastomer Copolymers	9011-17-0	VDF, HFP
	Fluoroelastomer Terpolymers	25190-89-0	TFE, HFP, VDF
	Base resistant elastomers	54675-89-7, 27029-05-6	TFE, VDF, P TFE, P
	Perfluoroelastomers	26425-79-6	TFE, PMVE
	CTFE elastomers	9010-75-7	CTFE, VFD
	Low temperature elastomers	CBI	TFE, VDF
Composite 4 - Aqueous Dispersions	PTFE	9002-84-0	TFE
	FEP	25067-11-2	TFE, HFP
	PFA	26655-00-5 31784-04-0	TFE, PPVE TFE, PEVE
	THV	25190-89-0	TFE, HFP, VDF

Confidential business information (CBI) regarding the chemical identity of Low temperature elastomers has previously been submitted to EPA under separate cover.

APPENDIX A.4

PREPARATION OF COMPOSITES TO BE TESTED

[NOTE: Drafting Committee discussions are continuing to finalize this section]

APPENDIX A.4

3. Preparation of Fluoropolymer Composites

3.1 Approach

A composite mixture of representative fluoropolymers, as solids, will be prepared for each of the four test substance composites identified in Table A-1.

The polymer samples will be first quality product polymer, substantially free of inorganic constituents. Each sample will be from a representative grade for each applicable fluoropolymer type from each applicable company.

A hypothetical example for Composite 2 in Table A-2 below shows how the composites will be assembled. In this example with 4 types across 4 companies, there are 11 x's. Hence, composite Z would be made up of 11 equal proportions of the materials indicated with an x.

Table A-2. Example for Compositing Across Companies & Types

Test Substance	Fluoropolymer Type	Company A	Company B	Company C	Company D
Composite Z	Type 1		x	x	x
	Type 2	x	x	x	x
	Type 3			x	
	Type 4	x	x	x	

For Composite 1 - PTFE resin (where there is a single fluoropolymer type), a representative sample of PTFE resin from each company producing PTFE resin will be mixed together in equal proportions across applicable companies to form the Composite 1 - PTFE resin.

3.2 Preparation

Representative samples of each component from each applicable company for each composite will be sent to the laboratory(ies) in packaging customarily used for product sample packaging or in polyethylene, polypropylene, or glass containers.

Each composite will be prepared under laboratory conditions designed to prevent cross-contamination and designed to assure solids temperatures less than or equal to 60 °C.

Following preparation of each composite, the composite will be placed in a polyethylene, polypropylene, or glass container.

3.2.1 Composite 1

PTFE resin is available in powder form. Equal weights of PTFE resin powder samples across applicable companies will be mixed together in dry form to yield Composite 1.

3.2.2 Composite 2

FEP, PFA, THV, ETFE, and HTE dry melt resins are available in pellet form. Each component of Composite 2 will be size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 3.1) will be mixed together in dry form to yield Composite 2.

A sample of polyethylene pellets will be size-reduced using the same technique and equipment to provide a blank. The resulting polyethylene powder will be archived.

[text on size reduction blank under further development]

3.2.3 Composite 3

Fluoroelastomers are available in slab, lump, or sheet form. Composite 3 will be prepared following one of the following approaches:

a) Equal weights of each component (following the approach in example for Composite Z in Section 3.1) will be mixed on a rubber mill to produce a homogenous slab of preset thickness to yield Composite 3.

Or

b) Each component of Composite 3 will be cyrogenically cooled (to make the elastomers brittle) and size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 3.1) will be mixed together in dry form to yield Composite 3.

A sample of non-fluorinated synthetic rubber will be size-reduced using the same technique and equipment to provide a blank. The resulting non-fluorinated rubber sample will be archived.

[text on size reduction blank under further development]

3.2.4 Composite 4

Aqueous dispersions of PTFE, FEP, PFA, and THV are available as dispersions containing 20 to 60% fluoropolymer solids by weight. Composite 4 will be prepared following one of the following approaches:

a. Equal weights (on a dry solids basis) of each component in aqueous dispersion form (following the approach in example for Composite Z in Section 3.1) will be mixed together in liquid form. Solids will be separated from the resulting liquid composite to yield low water content (i.e., drip free) fine solids.

Or

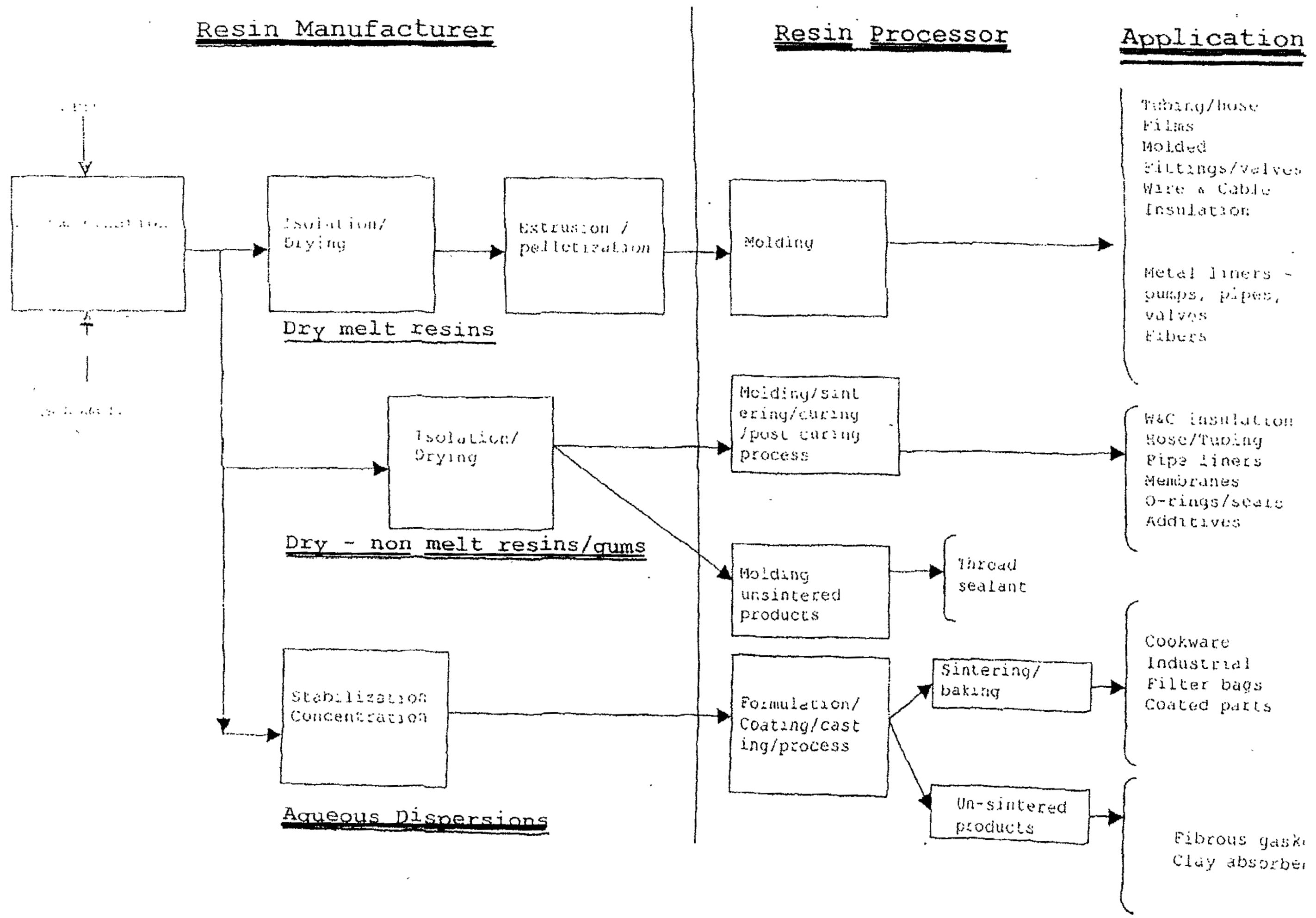
b) Solids will be separated from liquid for each component of Composite 4 to yield low water content (i.e., drip free) fine solids for each component. Equal weights of the solids form of each component (following the approach in the example for Composite Z in Section 3.1) will be mixed together to yield Composite 4.

3.3 Verification

In order to assure that composite samples in this testing program have been made up of clearly identified materials, the preparation of the composites will include formal Chain of Custody procedures. A chain of custody form will be included with each component material going into the composite to show the identity of the component material and each transfer of custody from its point of origination to preparation of the composite. For documentation, the laboratory preparing a given composite will generate a report to be submitted to EPA as CBI along with a sanitized version for the public record from which CBI has been removed.

Once prepared, each composite will be accompanied by a new chain of custody until it reaches the incineration testing facility.

Figure A-1. Fluoropolymer Industry Overview



Note to the Interested Parties:

The following pages provide a compilation of the most recent (as of 1/21/04) documents available to form a draft of the Appendices to be attached to the draft ECA documents for Laboratory-Scale Incineration Testing. Appendices B - G will be common to both incineration ECAs. The Drafting Committee has included the following identifier to show where additional work is needed:

[NOTE: Drafting Committee discussions are continuing to finalize this section]

APPENDIX B

TEST STANDARDS

B.1 ASTM E 18680-02 Loss-on-Drying by Thermogravimetry

APPENDIX B.1
GUIDELINE FOR THERMOGRAVIMETRIC ANALYSIS

ASTM E 1868-02 "Standard Test Method for Loss-On-Drying by Thermogravimetry" will be used as the guideline for conducting the analysis described in Appendix C.2.3 with the following modifications for this testing program:

Section	Modification
2.1	• Standard practices at the University of Dayton Research Institute (UDRI) may be used as references throughout the standard in place of the ASTM standards noted in this section.
4.1	• The loss-on-drying value specified in the second through fifth sentences of this section will not be recorded.
7.1.3	• The programming rate of the furnace will be set at 10 to 25°C/min, rather than 5°C/min. Pursuant to section 11.6, the temperature program rate will be documented in the report. • The isothermal temperature within the range of 25 to 1000°C will be maintained ±3°C, rather than ±2°C.
7.1.4	• The specimen atmosphere control system will be capable of supplying dry air in addition to "inert dry gas (usually purified grade nitrogen)".
7.1.7	• The temperature program rate will be set at 10 to 25°C/min, rather than 5°C/min. Pursuant to section 11.6, the temperature program rate will be documented in the report. • The temperature program rate will be controlled to within the range of ±3°C/min, rather than ±0.1°C/min. • Within the range of 25 to 1000°C, the isothermal temperature will be maintained within ±3°C, rather than ±2°C.
11.4	• The mass of the test specimen noted in the first sentence of this section will be 0.005 to 5 mg, rather than 10±1 mg (i.e., 9 to 11 mg).
11.6	• The test specimen heating rate will be set at 10 to 25°C/min, rather than 5°C/min noted in the first sentence of this section. Pursuant to section 11.6, the temperature program rate will be documented in the report.
11.9	• Termination criteria will follow Test Method A as

	outlined in section 11.10.1.
11.10.1	• The "fixed period of test time" mentioned in this section will be set at 5 min.
11.10.1.1	• Loss-on-drying values will not be recorded.
12.1	• The loss-on-drying value will not be calculated.
13.1.1	• The "identification and description of the material being tested" will be consistent with the information known to the analyst.
13.1.5	• The loss-on-drying value will not be included in the report.
14.2	• This section is not applicable because the Test Method A termination criteria will be used.

Reference

ASTM E 1868-02 "Standard Test Method for Loss-On-Drying by Thermogravimetry", ASTM International. For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

APPENDIX C

PROTOCOLS AS TEST STANDARDS

C.1 Transport Efficiency Testing
C.2 Incineration Testing
    C.2.1 Elemental Analysis
    C.2.2 Combustion Stoichiometry
    C.2.3 Thermogravimetric Analysis
    C.2.4 Combustion Testing
    C.2.5 Study Reporting

APPENDIX C.1
PFOA TRANSPORT TESTING

C.1.1 Significance

Testing will be performed to verify that potential PFOA emissions from the combustion testing described in Appendix C.2 can be quantitatively transported from the high temperature reactor into the exhaust gas sampling apparatus (aqueous solution bubblers).

Acceptable PFOA transport will be demonstrated if the transport efficiency (as computed in one or more of the formulas below) is greater than or equal to 70%.

C.1.2 Experimental Plan

C.1.2.1 Base Plan

Transport of PFOA across the laboratory-scale thermal reactor system described in Appendix C.2.4 and into the exhaust gas bubblers described in Appendix D.1 will be quantitatively determined as an indication of transport from the high temperature reactor into the bubblers.

A PFOA standard of known purity greater than or equal to 97% will be gasified at 150 to 250 °C (based on thermogravimetric analysis of PFOA) with transfer line and reactor temperatures 0 to 100 °C higher than the gasification temperature.

Three replicate transport efficiency test runs will be conducted. A minimum of one blank run will be conducted prior to each transport efficiency test run.

The sample size of the PFOA standard to be gasified will be less than 5 mg. The reactor exhaust gas will be collected into bubbler aqueous solution as described in Appendix D.1 (including an HPLC water rinse of the flexible tubing [used to connect the thermal reactor system and the bubbler assembly] into the aqueous solution composite), which will be analyzed for PFOA as described in Appendix D.2. In order to provide a second way of demonstrating quantitative transport, this aqueous solution composite will also be analyzed for total fluorine as described in Appendix D.3. (Testing for total fluorine is included due to possibility of thermal degradation of PFOA under transport test conditions.) Therefore, for this transport testing the amount of PFOA fed to the thermal reactor system will be sufficiently high to assure that the total fluorine input to the thermal reactor system will be greater than 140% of the mass corresponding to the limit of quantitation (LOQ) for total fluorine in the aqueous solution composite. (The LOQ for total fluorine in aqueous solution is much higher than the LOQ for PFOA in aqueous solution.)

The amount of PFOA and total fluorine in the thermal reactor system exhaust gas will be determined via analysis of the aqueous solution composite as noted above.

The amount of PFOA fed to the thermal reactor system will be known based on measurement prior to gasification and will be verified by weighing the pyroprobe insert cartridge before and after each test run. The amount of fluorine input to the system will be calculated from the amount of PFOA fed, the known purity of the PFOA, and the known fluorine fraction of the PFOA standard.

PFOA transport efficiency (TE) as a percentage will be computed as follows:

$$\%\ \text{PFOA TE} = \frac{\text{mass of PFOA in aqueous solution composite}}{\text{mass of PFOA fed to thermal reactor system}} * 100 \qquad (1)$$

Total fluorine (TF) transport efficiency as a percentage will be computed as follows:

$$\%\ \text{Total F TE} = \frac{\text{mass of total F in aqueous solution composite}}{\text{mass of total F fed to thermal reactor system}} * 100 \qquad (2)$$

C.1.2.2 Contingent Testing

If the transport efficiencies for both PFOA (equation 1) and total fluorine (equation 2) are less than or equal to 70%, then additional work will be performed. This additional work will be performedin a step-wise fashion to determine if consideration of one or more of the following procedural revisions enables achievement of 70% transport efficiency as follows:

Step 1. The flexible tubing between the thermal reactor system and the bubbler assembly from the experiment described in Section C.1.2.1 would be quantitatively rinsed with methanol. This methanol rinsate would be analyzed for PFOA (as described in

Appendix D.2) and/or for total fluorine (as described in Appendix D.3). Revised transport efficiency (TE) as a percentage for PFOA (equation 3) and/or total fluorine (equation 4) would be computed by including the mass of analyte in the methanol rinse in the numerator as follows:

$$\% \text{ PFOA TE} = \frac{\text{mass}_{PFOA} \text{ out}}{\text{mass}_{PFOA} \text{ in}} * 100 \qquad (3)$$

where mass_{PFOA} out = mass of PFOA in bubbler aqueous solution composite + mass of PFOA in methanol rinse

and mass_{PFOA} in = mass of PFOA fed to thermal reactor system

$$\% \text{ Total F TE} = \frac{\text{mass}_{total\ F} \text{ out}}{\text{mass}_{total\ F} \text{ in}} * 100 \qquad (4)$$

where $\text{mass}_{total\ F}$ out = mass of total F in bubbler aqueous solution composite + mass of total F in methanol rinse

and $\text{mass}_{total\ F}$ in = calculated mass of total F in PFOA fed to thermal reactor system

Step 2 (if necessary) The experiment described in Section C.1.2.1 would be repeated with reagent(s) (e.g. NaOH) added to the bubbler aqueous solution to determine if reagent addition enhances analyte absorption, thereby improving transport efficiency. Transport efficiency would be calculated using equation (1) and/or (2) above. The impact of reagent addition on LOQ for PFOA analysis described in Appendix D.2 would be determined.

C.1.3 Reporting of Results

Following completion of PFOA transport testing as described in this appendix and prior to beginning incineration testing described in Appendix C.2, a letter report will be submitted to EPA with the transport efficiency result(s) and indication of what contingent testing, if any, was performed.

If Appendix C.2 incineration testing is performed, the detailed results of Appendix C.1 transport testing will be included in the final report for Appendix C.2 incineration testing. If Appendix C.2 incineration testing is not performed, the detailed results of Appendix C.1 transport testing will be provided in a test report for Appendix C.1 transport testing.

APPENDIX C.2
INCINERATION TESTING

C.2.1 Elemental Analysis

C.2.1.1 Introduction

Elemental analysis as described in Section C.2.1 will be performed for each test substance composite to aid in preparation for combustion testing described in Section C.2.4.

As Kissa (1998) points out, technique strongly affects analytical results for fluorinated organic compounds such as fluorinated surfactants and fluorinated polymers due to the strength of the carbon-fluorine bond:

> Fluorine in organic compounds is usually determined by converting organic fluorine to an inorganic fluoride. Various combustion methods are routinely used for this purpose. However, the carbon-fluorine bond is exceptionally strong, and extremely vigorous conditions are needed for a quantitative mineralization. Conventional combustion conditions used for the determination of carbon and hydrogen in nonfluorinated organic compounds are not adequate for a quantitative analysis of fluorinated surfactants.

Therefore, total fluorine analysis will be performed using "extremely vigorous conditions" as described in Section C.2.1.2, and the commercially available conventional technique used for empirical determination of carbon and hydrogen content (described in Section C.2.1.3) will provide estimated values.

C.2.1.2 Total Fluorine

Each test substance composite will be characterized via analysis of total fluorine content.

Based on manufacturing process knowledge, the levels of total fluorine in the components of test substance composites are orders of magnitude higher than the potential trace level of inorganic fluoride in these materials. Therefore, for this test program, the total organic fluorine value for each test substance composite will be considered to be the same as the total fluorine value.

Total fluorine content will be measured via the Wickbold Torch method; see Appendix D.3.

C.2.1.3 Carbon and Hydrogen

In order to provide information for stoichiometric calculations in Section C.2.2, the carbon and hydrogen content of each test substance composite is needed. Based on manufacturing process knowledge of the polymers in this program, levels of sulfur, and nitrogen are expected to be less than 0.1% and to thereby have negligible effect on stoichiometric calculations.

C.2.1.3.1 Theoretical Determination

Where the elemental composition of a test substance composite is known from the identity of the components in a given composite, the carbon and hydrogen content of the test substance composite can be calculated.

For example, where each of the components of a test substance composite are polytetrafluoroethylene (PTFE), the carbon and hydrogen can be determined knowing the molecular formula for PTFE is $(C_2F_4)_n$ as follows:

	number	atomic weight	weight %
carbon (C)	2	12	24
hydrogen (H)	0	1	0
fluorine (F)	4	19	76
total			100

C.2.1.3.2 Empirical Determination

Where compositional information on carbon and hydrogen content is not known from the identity of the components in a given composite, each such test substance composite will be analyzed for carbon and hydrogen.

As noted in Section C.2.1.1, empirical determination of carbon in test substance composites via commercially available conventional techniques is expected to underestimate the carbon content of the test substance composites due to the strength of the carbon-fluorine bond. Similarly, empirical determination of hydrogen in test substance composites via commercially available conventional techniques is expected to overestimate the hydrogen content of the test substance composites.

The carbon content of the test substance composite can be measured by determining the carbon dioxide (CO_2) generated by the oxidation of the sample. This oxidation may be accomplished by high temperature combustion, catalytic combustion, or wet chemical oxidation. The CO_2 is measured directly by an infrared detector or a thermal conductivity detector, via absorption into a suitable solution (e.g., potassium hydroxide) and gravimetric determination, or by conversion to methane for measurement via a flame ionization detector.

The hydrogen content of the sample can be determined by difference with knowledge of the fluorine content and carbon content of the sample where the moisture content and chlorine content of the sample are negligible or known. Alternatively, the hydrogen content of the sample is measured by determining the water generated by high temperature combustion of the sample. Measurement of water in the combustion gas for this analysis may be accomplished by techniques such as use of an infrared detector or absorption on a dessicant with gravimetric determination. With empirical hydrogen determination, it is important to correct for the water in the combustion gas attributable to the moisture content in the sample to obtain the hydrogen content of the sample; see Section C.2.1.4.

Manufacturing process knowledge of the polymers will be used to review the elemental analysis results and to form the basis for interpreting non-detects. For example, if the hydrogen analytical result for a perfluorinated polymer is less than a quantitation limit of 0.1%, then the analytical result will be replaced with 0.

C.2.1.4 Moisture

Where preparation (as described in Appendix A.4) for a given test substance composite has involved dewatering, the moisture (or solids) content of each such test substance composite will be determined in order to provide a dry basis for calculations as needed.

Moisture is determined by measuring the loss of weight of the sample when heated under controlled conditions. A representative sample is weighed and placed in a crucible (or dish) and evaporated to dryness in an air or nitrogen atmosphere at a defined temperature setpoint (e.g., 103 °C to 105 °C) in the range of 100 °C to 125 °C.

The moisture value is calculated as the loss in weight (difference between the starting weight of sample and the final weight of sample) divided by the starting weight of sample. Similarly, a solids value can be calculated as the final weight of sample divided by the starting weight of sample.

C.2.2 Combustion Stoichiometry

Combustion stoichiometry calculations as described in Section C.2.2 will be performed to aid in preparation for combustion testing described in Section C.2.4

First, the weight percent values from Section C.2.1 are converted to molar quantities on a dry basis.

Second, based on Chapter 3 of *Combustion Fundamentals for Waste Incineration* (American Society of Mechanical Engineers, 1974), the reaction products for these molar quantities are calculated assuming complete combustion with the following rules:

a) All carbon (C) in feed converts to carbon dioxide (CO_2)

$$C + O_2 \rightarrow CO_2$$

b) All sulfur (S) in feed converts to sulfur dioxide (SO_2)

$$S + O_2 \rightarrow SO_2$$

c) The halogens (Cl, F) in feed convert to hydrogen halides

$$H_2 + Cl_2 \rightarrow 2HCl$$
$$H_2 + F_2 \rightarrow 2HF$$

d) Hydrogen (H) present in feed in excess of that required to yield products in item c) above will be converted to water

$$2H_2 + O_2 \rightarrow 2H_2O$$

e) Nitrogen (N) from feed or air is emitted as molecular nitrogen

$$N_2 \rightarrow N_2$$

Third, with these rules, the balanced chemical reaction for combustion of a compound can be written.

For example, the resulting reaction equation for a hydrocarbon like methane (CH_4) is

$$CH_4 + 2\ O_2 \rightarrow CO_2 + 2H_2O$$

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Note that the term feed in the preceding rules (a through e) includes both material being combusted and the fuel source of hydrogen such as methane or methanol. Additionally, stoichiometric calculations as described above presume that the compounds undergoing combustion are essentially free of inorganic constituents.

These calculations provide the theoretical amount of oxygen needed for the overall combustion reaction for the feed based on the available information used in the calculations. The initial estimate for the amount of oxygen to be used in combustion testing will be determined from this theoretical amount with adjustments for target oxygen level in thermal reactor system exhaust gas. The actual amount of oxygen to be used in combustion testing will be based oxygen monitoring described in Section C.2.4.

These stoichiometric calculations will also be used as needed to initially estimate and adjust experimental conditions for combustion testing in Section C.2.4.

C.2.3 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) will be conducted to determine the temperature range required for gasification of each test substance composite. TGA will be conducted in flowing air from room temperature to 1000°C as described in Appendix B.1.

The TGA weight-loss profile for each test substance composite will be evaluated to determine the temperature at which the weight loss reaches a final asymptote across the temperature range investigated. This temperature corresponds to the point at which no further gasification (under test conditions) occurs for the material and will be considered the temperature for complete gasification of the material.

C.2.4 Combustion Testing

C.2.4.1 Test Objective

The objective of the testing program described in Appendix C.2 is to assess the potential for waste incineration of each test substance composite to emit PFOA, based on quantitative determination of potential exhaust gas levels of PFOA from laboratory-scale combustion testing under conditions representative of typical municipal waste combustor operations in the U.S.

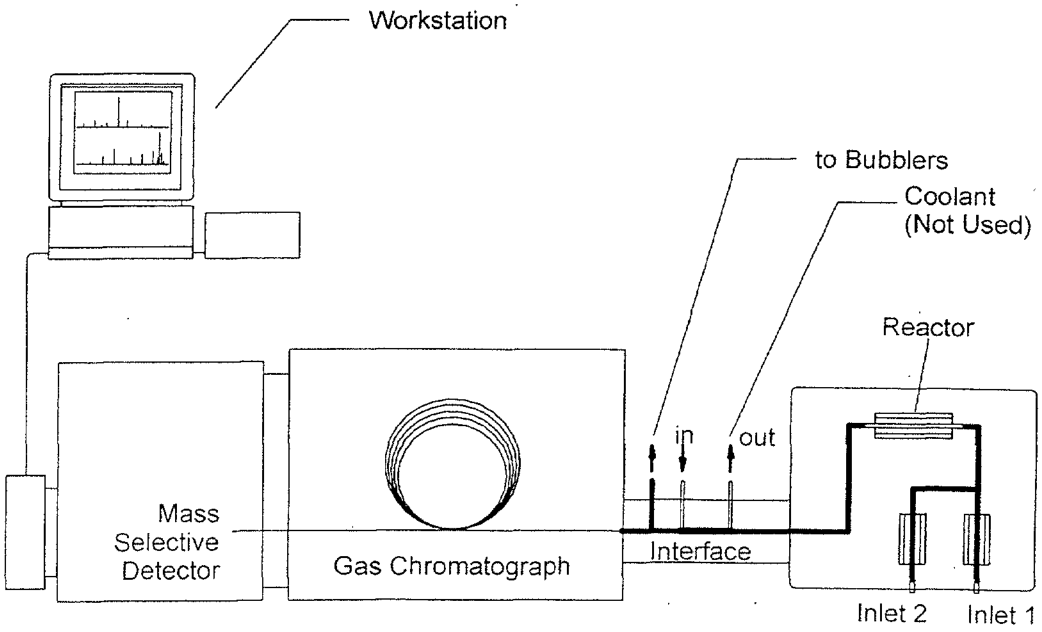
C.2.4.2 Experimental Apparatus

Combustion testing will make use of the Advanced Thermal Reactor System (ATRS) at the University of Dayton Research Institute (UDRI). The ATRS is a laboratory-scale, non-flame, batch-charged, continuous flow thermal reactor system. The use of this non-flame thermal reactor system gives a conservative representation of full-scale waste incineration prior to air pollution controls.

In the ATRS, the test sample is gasified and transported to a high temperature reactor. In the high temperature reactor, the sample vapors are subjected to controlled conditions for residence time and temperature. As described in Sections C.2.4.5 and C.2.4.6, combustion products will be monitored or collected for quantitative analysis.

A schematic of the ATRS as configured for this test program is shown in Figure C.2-1.

Figure C.2-1. Schematic of ATRS for this Test Program



The ATRS consists of a reactor assembly and in-line gas chromatograph/detector system connected via an interface.

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The reactor assembly consists of a thermally insulated enclosure housing the sample introduction, reactor, and transfer line systems.

Sample introduction for solid materials (Inlet 1) employs a pyroprobe, a device designed to gasify samples by heating them at a fixed rate. The main gas flow will also be fed via Inlet 1, and Inlet 2 will be used to feed supplemental flow.

During combustion tests, the transfer line between the pyroprobe and the reactor is heated and maintained above 200 °C. The reactor is housed within its own small tube furnace and may be independently heated to as high as 1100 °C. (Actual conditions for this test program are presented in Section C.2.4.3.) The transfer line from the reactor to the interface is heat traced to greater than 200 °C to prevent cool regions where reactor products could otherwise be lost through condensation.

The interface routes the combustion exhaust gas to the in-line gas chromatograph (GC) and mass selective detector (MSD) or to sample collection for off-line analysis. For combustion testing in this test program, the interface will also be maintained above 200 °C. Exhaust gas monitoring for this program is described in Section C.2.4.5.

C.2.4.3 Combustion Test Experimental Conditions

Each test substance composite will be subjected to laboratory-scale incineration using the experimental apparatus described in Section C.2.4.2.

C.2.4.3.1 Combustion Air

Synthetic air (mixture of 21% oxygen and 79% nitrogen) will be used in place of compressed air to prevent potential interference in the experimental system due to background levels of CO_2 in compressed air.

C.2.4.3.2 Fuel

Methanol will be used, as needed, as a supplemental fuel to ensure the presence of sufficient hydrogen to convert fluorine to hydrogen fluoride (HF) and chlorine to hydrogen chloride (HCl).

As noted in *Municipal Solid Waste in the United States: 2000 Facts and Figures* (EPA, 2002), paper and paper products (made from wood) make up the largest component of municipal solid waste (MSW). The sum of paper and paper products with wood in MSW makes up over 30% of MSW.

During the 19th century, methanol was produced from wood and was known as wood alcohol. Therefore, methanol can be used in this experimental program as a surrogate for the paper and wood fraction of MSW.

C.2.4.3.3 Operating Conditions

The target operating conditions for the high temperature reactor during the combustion tests for each test substance composite identified in Appendix A.3 are presented in Table C.2-1.

Table C.2-1. Combustion Test Target Operating Conditions

Temperature	1000 °C
Residence Time	2 sec
O_2 concentration in exhaust gas	10%
H_2O concentration in exhaust gas	15%
Number of replicate runs	3

These conditions are conservatively representative of typical furnace operating conditions of municipal waste combustors (MWCs) and of typical secondary chamber operating temperatures for medical waste incinerators in the U.S. See Appendix D.4 for supporting information.

Temperature and residence time values in Table C.2-2 will be fixed setpoints for these experiments. The temperature of the high temperature reactor will be controlled within ±10 °C to assure isothermal operation.

The amount of each test substance composite fed to the ATRS in this testing program will be a measured amount less than 5 mg. The actual amount fed, gasification rate (determined from TGA), air supply, and fuel supply will be adjusted to assure that the oxygen level in the exhaust will be greater than or equal to the concentration in Table C.2-1 throughout each test to be representative of typical MWC conditions. The fuel supply and air supply will also be adjusted as needed to approach the target H_2O concentration in exhaust gas in Table C.2-1.

The pyroprobe section final temperature (at end of temperature ramp-up) will be 750 °C or as needed to assure this section is 50 to 100 °C above the highest temperature for complete gasification across the test substance composites as determined from the TGA results; see Section C.2.3. This is necessary to assure complete gasification of the sample of test substance composite and a common set of experimental conditions across the test materials during combustion testing.

C.2.4.3.4 Blanks

A minimum of one thermal blank will be run prior to each set of three combustion test runs for a given test substance composite. Each thermal blank run will be at the corresponding combustion test conditions with all feeds except for the test substance.

C.2.4.4 Process Monitoring

ATRS process parameters in Table C.2-2 will be monitored for each combustion test at key points during the test as noted in the table. Each combustion test will be a minimum of 5 minutes in duration. If the duration of a combustion test is greater than 15 minutes, each parameter in Table C.2-2 will be recorded at least once every 15 minutes.

Table C.2-2. Combustion Test Monitoring

Parameter	Key Time for Recording
Temperature-Reactor	Before & after gasification
Temperature-Transfer line	Before & after gasification
Temperature-Inlet 1	After gasification
Temperature-Inlet 2	Before & after gasification
Gas flow rate-Inlet 1	Before & after gasification
Gas flow rate-Inlet 2	Before & after gasification
Total Gas Flow rate	Before & after combustion test
Make-up Gas (He) Flow rate	Before & after combustion test
Pressure-Reactor	Before & after gasification

Temperature-Inlet 1 will be recorded at the end of the temperature ramp-up for gasification to monitor the pyroprobe final temperature.

The flow rate of the exhaust gas routed to the bubblers (see Section C.2.4.5.2) will be determined based on the flow measurements listed in Table C.2-2.

The amount of material fed to the system will be verified by weighing the pyroprobe insert cartridge before and after each experiment.

Exhaust gas monitoring is described in Section C.2.4.5.

C.2.4.5 Exhaust Gas Monitoring

Combustion exhaust gas will be continuously monitored for oxygen during each combustion test via in-line MSD or via an oxygen monitor. CO_2 in exhaust gas will be monitored via in-line GC, in-line MSD, or a continuous monitor; or exhaust gas will be collected in Tedlar® bags for off-line analysis of CO_2. Carbon monoxide (CO) in exhaust gas will be monitored via in-line GC or a continuous monitor; or exhaust gas will be collected in Tedlar® bags for off-line analysis of CO. Tedlar® bag samples may be collected at the exit of the bubblers described in Section C.2.4.6.

C.2.4.6 Exhaust Gas Sampling

Gas samples for off-line analysis will be collected as described in Appendix D.1.

A minimum of 60 mL of bubbler aqueous solution composite is expected from each combustion test. Of this, a minimum of 45 mL will be directed to PFOA analysis, and the remainder will be directed to fluoride ion analysis.

C.2.4.7 Exhaust Gas Analysis

C.2.4.7.1 Fluoride Ion

A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section C.2.4.6 will be analyzed for fluoride ion via ion chromatography.

C.2.4.7.2 PFOA

A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section C.2.4.6 will be analyzed for PFOA via LC/MS/MS as described in Appendix D.2.

As described in Appendix D.2, composite bubbler aqueous solution sample results less than method detection limit (MDL) will be reported as not detected (ND), results between MDL and the limit of quantitation (LOQ) will be reported as not quantifiable (NQ), and numerical values will not be reported.

Due to background levels of PFOA, the analytical laboratory will only report numerical values for PFOA concentration in the aqueous solution greater than or equal to the LOQ. This is required to assure that the reported concentration value is attributable to the aqueous solution sample rather than to background.

C.2.5 Reporting of Results

C.2.5.1 Elemental Analysis Results

The results of elemental analysis for each test substance composite (as noted in Section C.2.1) will be reported. The laboratory reports will be included in an appendix to the final report for incineration testing (test report).

C.2.5.2 Combustion Stoichiometry Results

Combustion stoichiometry (as noted in Section C.2.2) calculations for each test substance composite will be included in an appendix to the test report.

C.2.5.3 TGA Results

The temperature for complete gasification and the TGA graphical results for each test substance composite (as noted in Section C.2.3) will be included in an appendix to the test report.

C.2.5.4 Combustion Test Results

C.2.5.4.1 Process Monitoring

Process monitoring data (as noted in Section C.2.4.4) recorded for each combustion test will be reported in tabular form.

C.2.5.4.2 Exhaust Gas Monitoring

Exhaust gas O_2, CO and CO_2 monitoring results will be reported as the integrated or average value for each combustion test. CO will be reported in terms of parts per million by volume (ppmv). O_2 and CO_2 will be reported in terms of percent by volume (%).

C.2.5.4.3 Exhaust Gas Analytical Results

Results of analyses noted in Section C.2.4.7 will be reported for each replicate of each combustion test.

The analytical result for each analyte in Section C.2.4.7 will be reported in terms of concentration (mass per volume) in the bubbler aqueous solution. For each analyte, this value will be used with the associated exhaust gas volume to compute an exhaust gas concentration and with the associated test substance mass to compute mass of analyte per mass of test substance composite.

C.2.5.4.3.1 Fluoride

Fluoride ion in the exhaust gas will be reported on the basis of mass of fluoride ion per mass of test substance composite. The corresponding hydrogen fluoride value for each will also be computed and reported for reference.

C.2.5.4.3.2 PFOA

PFOA results for the bubbler aqueous solution samples will be reported as described in Section C.2.4.7.2. PFOA results for associated blanks will also be reported.

If present in the bubbler aqueous solution at a concentration above the matrix-specific LOQ, PFOA in the exhaust gas will be reported on the basis of mass of PFOA per mass of test substance composite.

C.2.5.5 Release Assessment

In the event that PFOA is reported for the exhaust gas bubbler aqueous solution at a concentration above the LOQ for the three runs for a given test substance composite, a release assessment report for the subject material will be prepared following the outline in Appendix E.2 and will be included in the test report.

APPENDIX D

ATTACHMENTS AND REFERENCED MATERIALS

D.1 Exhaust Gas Sampling
D.2 PFOA Analysis Method
D.3 Wickbold Torch Method
D.4 Waste Incineration and Operation Conditions

APPENDIX D.1
EXHAUST GAS SAMPLING VIA BUBBLERS

Gas samples for off-line analysis will be collected from a vent line off the interface of the thermal reactor system described in Appendix C.2.4. Flexible (silicone or equivalent) tubing will connect the vent line and a set of bubblers.

Gas absorption via these bubblers will provide aqueous solution (of documented content) to analyze for prescribed parameters. Two to four bubblers (low pressure drop impingers) in series will be used. Each bubbler will contain a predetermined amount of aqueous solution, and the total amount of solution at the beginning of each test run will be a minimum of 55 mL. The temperature of the gas exiting the last bubbler will be monitored.

An additional bubbler (which is empty) will be added to the front end of this series of bubblers to serve as a knock-out pot if calculations or preliminary measurements indicate that greater than 10 mL of water will be produced during the testing for a given material.

Upon completion of sample collection, the amount in each bubbler will be weighed and recorded, and the contents of the bubblers will be composited for subsequent analysis. Additionally, the flexible tubing will be rinsed with 5 mL of HPLC water to collect potential condensate in the tubing; this rinsate will be combined with the bubbler composite prior to analysis.

Bubbler aqueous solution composites will be conveyed to analytical laboratory(ies) in polyethylene, polypropylene, or glass container(s).

APPENDIX D.2
PFOA ANALYSIS BY LC/MS/MS

D.2.1 Introduction

Samples to be analyzed for PFOA in this study will be subjected to Liquid Chromatography with Tandem Mass Spectrometry (LC/MS/MS) in accordance with "Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water Revision 1" (Exygen method) revised per the section-by-section comments listed in Section D.2.4 below. These revisions are necessary to adapt a method originally developed for liter quantity water samples to samples related to testing described in Appendix C.

The testing programs described in Appendix C are expected to generate samples of aqueous solution, methanol (e.g., as used for extraction or rinsing), and corresponding blanks. The expected sample size for aqueous solution samples (from exhaust gas bubbler sample collection) available for analysis via this method is approximately 50 mL.

D.2.2 Method Summary

PFOA is extracted from water using a disposable C_{18} solid phase extraction (SPE) cartridge. PFOA is eluted from the cartridge with methanol. Quantification of PFOA is accomplished by electrospray liquid chromatography/tandem mass spectrometry (LC/MS/MS) analysis.

D.2.3 Reporting

The target limit of quantitation (LOQ) for this study with this method is 50 ng/L based on prior work with water samples where an 8-fold concentration via extraction using C_{18} SPE cartridge has been demonstrated. The actual LOQ will be matrix dependent; for samples (e.g., methanol rinsate) where the 8-fold concentration cannot be performed, the target LOQ for this study is 400 ng/L.

Sections 4.5.4 and 5 of the Exygen method explain reporting for field samples such as bubbler aqueous solution composites, which are distinct from blanks and spikes, as follows:

> Field samples in which either no peaks or peaks less than the

> MDL are detected at the corresponding analyte retention time will be reported as ND (not detected). Samples in which peaks are detected at the corresponding analyte retention time that are less than the LOQ and greater than or equal to the MDL will be reported as NQ (not quantifiable).

Therefore, sample results less than method detection limit (MDL) will be reported as ND, and sample results between MDL and the limit of quantitation (LOQ) will be reported as NQ. Numerical values will not be reported for such samples. Only concentrations above the LOQ, where the reported concentration is attributable to the sample rather than to background, are reported with numerical values.

Additionally, if the PFOA anion is found in a sample at a concentration above the LOQ for the matrix but is less than 5 times the concentration found in the associated blank, the result will be flagged and treated as ND.

D.2.4 Study-Specific Comments on the Method

Section	Comment
1	• The concentration of PFOA found will be reported directly and the mathematical conversion for reporting as APFO mentioned in the 4th sentence of the 2nd paragraph will not be performed. • Since the 8-fold concentration described in the 2nd sentence 4th paragraph (which forms the basis for the LOQ in the 3rd paragraph and the MDL in the 4th paragraph) is dependent on having a minimum of 40 mL of aqueous sample amenable to extraction using the C_{18} SPE cartridge described in section 4.4 of the method, the LOQ and MDL in the method will be a factor of 8 higher than reported where less than 40 mL of sample is available or where the sample is not amenable to extraction using the C_{18} SPE cartridge described in section 4.4 of the method (e.g., methanol).
3.3 Note at top of page 8	• The note stating "Equivalent materials may be substituted for those specified in this method if they can be shown to produce satisfactory results" will not be used in the analysis for this testing program.
3.3 Notes,	• The following text will be used in place of Note 1 with respect to the PFOA analysis

Note 1	conducted for this testing program: In order to avoid contamination, the use of disposable labware (tubes, pipets, etc.) is required.
3.3 Notes, Note 4	• The following text will be used in place of Note 4 with respect to the PFOA analysis conducted for this testing program: Solvents (e.g., methanol) used for this analysis must be checked for the presence of contaminants by LC/MS/MS before use.
3.5 opening text prior to 3.5.1	• Where the available amount of sample is expected to be much less than 1 liter, insufficient sample is available to prepare the fortified matrix spikes described in the opening text of section 3.5. In this case, the analytical standards discussed in this opening text will be limited to two purposes since the third purpose (matrix spike) stated in <u>the method cannot be</u> done.
4.3, item b	• Where the available amount of sample is expected to be less than 80 mL (= 2 * 40), the replicate extraction noted in the first sentence of this item cannot be performed. • If the sample is not an aqueous sample amenable to extraction using the C_{18} SPE cartridge described in section 4.4 of the method, then section 4.4 is skipped such that the sample is analyzed directly. (Note: For such samples, the LOQ and MDL will be 8 times higher than the values quoted in the method.)
4.3, item c	• As noted in comment on section 3.5 opening text above, fortified matrix spikes will not be prepared when the available amount of sample is much less than 1 liter. • Where the available amount of sample is expected to be less than 80 mL (= 2 * 40), the conditional repeat fortification and extraction described in the third sentence of this item cannot be performed.
4.4	• Extraction using the C_{18} SPE cartridge requires a suitable aqueous sample. This extraction and the corresponding 8-fold concentration pointed out in the NOTE at the end of this section cannot be performed on non-aqueous

	(e.g., methanol) samples.
4.4, item 1	• In order to measure out the 40 mL mentioned in this item, it is necessary to have at least 45 mL of sample to enable pipet transfer.
4.5.4, item g	• A storage stability study for PFOA in water performed independently of the development of the method indicates that PFOA may be stored in glass, polystyrene, polypropylene, or polyethylene containers without measurable degradation for up to 68 days prior to extraction. Therefore, the total holding time between sample collection and analysis for aqueous PFOA samples in this study may exceed the 14 day limit noted in the first sentence of this item provided that the sample is not held for greater than 68 days unless additional storage stability testing justifies a longer hold time.
4.6, item 3	• As noted in comment on section 3.5 opening text above, fortified matrix spikes will not be prepared when the available amount of sample is much less than 1 liter. In this case, acceptance criteria for matrix spike recoveries will not be considered.
5, item c	• The calculation in equation 3 in this section will not be performed since it is not necessary to convert the PFOA analytical results to APFO for this study.

D.2.5 Reference

Flaherty, J. and K. Risha, "Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water Revision 1", Exygen Method No. 01M-008-046 Revision 1, January 2003. (EPA Docket ID OPPT-2003-0012-0040)

APPENDIX D.3
WICKBOLD TORCH METHOD FOR TOTAL FLUORINE

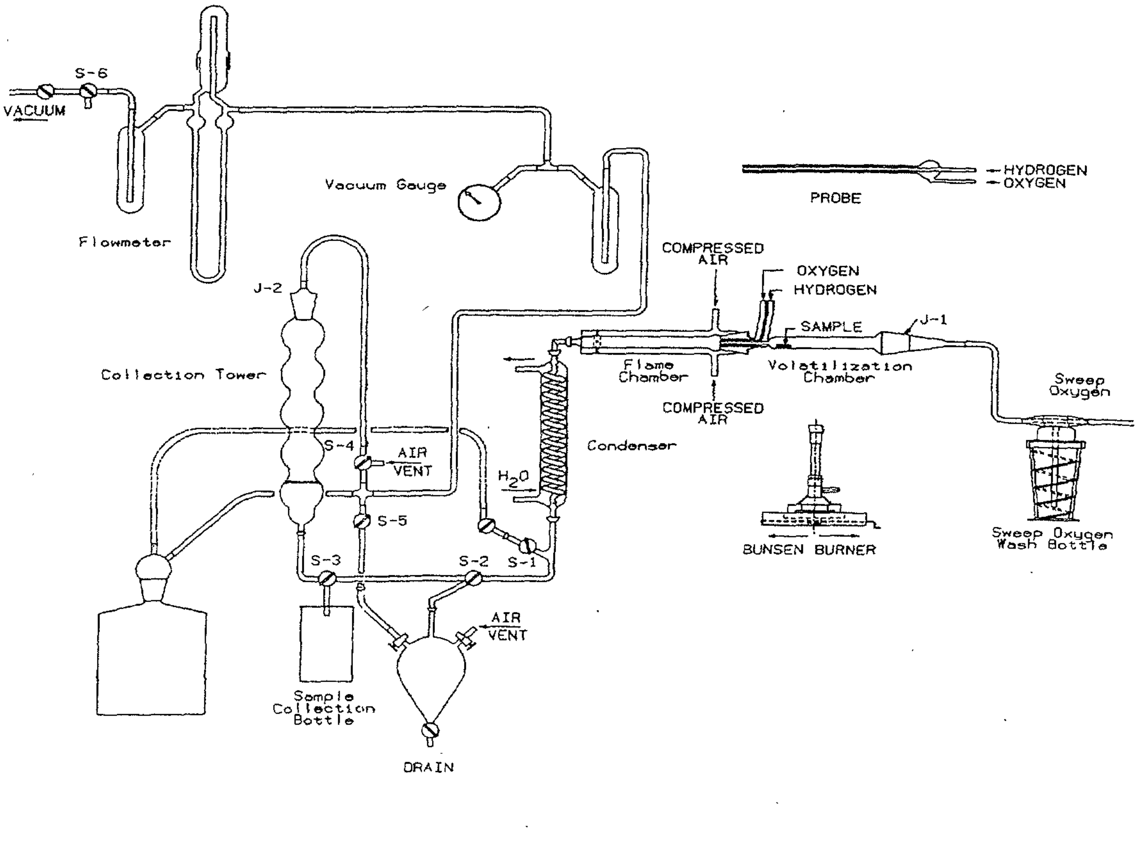
D.3.1 Introduction

"The carbon-fluorine bond is exceptionally strong, and extremely vigorous conditions are needed for quantitative" analysis of fluorine in organic compounds. (Kissa, 1998) The "most vigorous" technique for measurement of fluorine in organic compounds is "combustion in an oxyhydrogen flame" referred to as the Wickbold torch. (Kissa, 1998)

D.3.2 Apparatus

A typical configuration for the Wickbold oxyhydrogen torch apparatus as described by Sweetser (1956) is shown in Figure D.3-1.

Figure D.3-1. Wickbold Oxyhydrogen Torch Apparatus



D.3.3 Method Description

The sample size for the standard sample boat is up to 20 mg for a solid or up to 5 mL for a liquid.

With the oxyhydrogen torch in operation, the sample is pyrolyzed or vaporized with a Bunsen burner moving on a rail below the volatilization chamber. The vapors and pyrolysis products are swept through the oxygen-hydrogen flame chamber operating at up to approximately 2000 °C to mineralize the fluorine in the sample to fluoride ion. The resulting fluoride ion is absorbed in the collection tower containing water or an alkaline solution.

The absorbed fluoride ion is measured via fluoride ion-selective electrode or ion chromatography.

The reported limit of quantitation for total fluorine via the Wickbold Torch method is 0.5 ppm (0.5 mg/kg). The accuracy of this method for determination of total fluorine in fluorinated polymers is exemplified by total fluorine values of 75.35% to 75.84% for PTFE with known total fluorine content of 76.0%. (Sweetser, 1956)

D.3.4 Safety Considerations

Use of hydrogen presents a potential fire and explosion hazard. Use of oxygen presents a potential fire hazard. Safe operation of the oxyhydrogen torch is assured by the use of specialized equipment with shielding and elaborate safety devices by well-trained personnel at a qualified laboratory.

D.3.5 References

Kissa, E. "Analysis of Anionic Fluorinated Surfactants", Chapter 8 in Anionic Surfactants: Analytical Chemistry - 2nd Edition, Revised and Expanded, edited by John Cross. Marcel Dekker Surfactant Science Series, volume 73, 1998.

Sweetser, P. B. "Decomposition of Organic Fluorine Compounds by Wickbold Oxyhydrogen Flame Combustion Method", Analytical Chemistry, vol. 28, pp. 1766-1768, 1956.

APPENDIX D.4

Waste Incineration and Operation Conditions

[NOTE: Drafting Committee discussions are continuing to finalize this section]

[NOTE: DRAFTING COMMITTEE DISCUSSIONS ARE CONTINUING TO FINALIZE THIS SECTION]

APPENDIX A.4
PREPARATION OF FLUOROTELOMER-BASED POLYMER COMPOSITES

4.1 Assembly of Components

For each telomer-based polymeric product (TBPP) component for each test substance composite described in Appendix A.1, each company will collect a minimum of 100 mL of first, quality production of a representative grade of TBPP. Each company will send a minimum of 25 mL of each such TBPP component to a facility designated by the Telomer Research Program (TRP) and store the remainder of each such TBPP component under conditions at or below ambient temperature for a period of 5 years. Both parts will be contained in new, unused packaging customarily used for product sample packaging or in new, unused polyethylene, polypropylene, or glass container(s).

Transmission of TBPP components for test substance composite preparation in this program will include formal Chain of Custody procedures. For each TBPP component for each test substance composite, each company will assign a unique non-CBI identifying name (e.g., unique generic name) and identify which composite the component is to go into. This name and the identity of the composite it is to go into will be used as the "sample description" on the Chain of Custody form used when conveying TBPP component(s) to the compositing laboratory. The Chain of Custody form used when conveying TBPP component(s) to the compositing laboratory will also distinguish among the TRP member companies to verify that each company contributes to each applicable composite.

The TRP-designated facility assembling the components may be the compositing laboratory or may be a single common alternate facility. If such an alternate facility is used, then new Chain of Custody form(s) will be prepared, as needed to remove CBI while assuring component distinction, to accompany the TBPP component to the compositing laboratory.

The deadline for each company to submit its TBPP components to the TRP-designated facility is shown in Table of the ECA.

4.2 Preparation

The TBPPs described in Appendix A.1 are aqueous dispersions with nominally 20% solids, which contain the fluorotelomer based polymer (FTBP). Each test substance will be an FTBP solids composite following dewatering and will be prepared as described in Section 4.2.1 or as described in Section 4.2.2 below.

Composite preparation will be conducted under laboratory conditions designed to prevent cross-contamination and designed to assure solids temperatures less than 60 °C.

The telomer product solids composites will be substantially free of inorganic constituents.

Following preparation of each composite, each composite will be placed in a polyethylene, polypropylene, or glass container and will be accompanied by a new Chain of Custody (for the composite(s)) until each composite reaches the incineration testing facility.

4.2.1 Mixing Followed by Dewatering

The composite preparation sequence via mixing followed by dewatering is follows:

1. For each composite, the relevant TBPP components will be gathered.

2. A portion of each of these TBPP liquids will be analyzed to determine the amount of FTBP solids via measurement of Total Fluorine as described in Appendix D.3. The moisture content of a portion of each TBPP liquid will be determined as described in Appendix C.2.1.4.

3. The amount of each component TBPP liquid to go into a given composite will be established based on the Total Fluorine result from step 2 to assure that the FTBP solids of each component into a given composite will be present in equal proportions (on a Total Fluorine basis).

NOTE: — [EXAMPLE CALCULATION TO BE DEVELOPED]

4. For each composite, the component TBPP liquids will be mixed according to the amounts from step 3 to form the composite as a liquid.

5. For each composite as a liquid, the liquid will be

spread into sufficiently large aluminum pan(s). The material in the pan(s) will be dewatered via evaporation at ambient conditions (thereby assuring solids temperature less than 60 °C) in a laboratory hood (away from other potential sources of PFOA) for two days until the material is visibly free of excess water (i.e., visibly drip free). (A small amount of residual moisture is expected to be remaining in the dewatered material.)

6. The dewatered FTBP solids will be treated with liquid nitrogen as necessary to allow for easy release from the aluminum pan(s). The material will be transferred to a mortar and pestle and ground using liquid nitrogen as necessary to produce visibly consistent solids size.

4.2.2 Dewatering Followed by Mixing

The composite preparation sequence via mixing followed by dewatering is follows:

1. For each composite, the relevant TBPP components will be gathered.

2. A portion of each of these TBPP liquids will be analyzed to determine the amount of FTBP solids via measurement of Total Fluorine as described in Appendix D.3. The moisture content of a portion of each TBPP liquid will be determined as described in Appendix C.2.1.4.

3. The amount of FTBP solids for each TBPP component to go into a given composite will be established based on the Total Fluorine result from step 2 to assure that the FTBP solids of each component into a given composite will be present in equal proportions (on a Total Fluorine basis). The result from step 2 for Total Fluorine also establishes the minimum amount of TBPP liquid for each component needed for subsequent preparation steps.

NOTE — [EXAMPLE CALCULATION TO BE DEVELOPED]

4. For each component in each composite, an amount of the TBPP liquid greater than or equal to the minimum amount of each TBPP liquid from step 3 will be spread into sufficiently large aluminum pan(s). The material in each pan will be dewatered via

evaporation at ambient conditions (thereby assuring solids temperature less than 60 °C) in a laboratory hood (away from other potential sources of PFOA) for two days until the material is visibly free of excess water (i.e., visibly drip free). (A small amount of residual moisture is expected to be remaining in the dewatered material.)

5. The dewatered FTBP solids will be treated with liquid nitrogen as necessary to allow for easy release from the aluminum pan(s). The material will be transferred to a mortar and pestle and ground using liquid nitrogen as necessary to produce visibly consistent solids size.

6. The dewatered FTBP solids from step 5 for each relevant component in the amount of FTBP solids based on the Total Fluorine result from step 2 will be mixed together to form each composite.

4.3 Verification

To verify adherence to Section 4.2, the laboratory preparing a given composite will generate a report describing how the composite was prepared. This report will be included in the final report for Phase II incineration testing.

The Total Fluorine content (as described in Appendix D.3) and the moisture content (as described in Appendix C.2.1.4) of each composite will be determined as noted in Appendix C.2.1. The Total Fluorine content of each composite on a dry basis will be computed and included in the report prepared by the compositing lab.

The weighted average Total Fluorine content of the components of each composite will be computed on a dry basis based on the results from step 2 above and included in the report prepared by the compositing lab.

If CBI is in the report of the compositing lab, both a CBI version and a sanitized version for the public record (from which CBI has been removed) will be prepared.

APPENDIX E

E.1 Outline for Interim Progress Reporting
E.2 Release Assessment Report

APPENDIX E.1

OUTLINE FOR INTERIM PROGRESS REPORTING

Title : Enforceable Consent Agreement for the Laboratory-Scale Incineration Testing of Fluoropolymers - Interim Report

OPPT Docket ID No: OPPT-2003- 0071

Date of Interim Report: [date]

This Report covers the period from [date] to [date]

1) List or description of significant ECA Test Program milestones during this period:

2) Description of difficulties : (If none indicate N/A)

3) Actions taken in response to difficulties: (If none indicate N/A)

APPENDIX E.2

OUTLINE FOR RELEASE ASSESSMENT REPORT

[NOTE: Drafting Committee discussions are continuing to finalize this section]

As described in Part VII.B, footnote 3, Table 1 footnote ??, and Appendix C.2.5.5 of this ECA, if the Phase II incineration testing identifies measurable levels of PFOA (where measurable PFOA is defined to be at or above the limit of quantitation (LOQ) as defined in Appendix D.2) resulting from the incineration testing for any or all of the test substance composites tested under this ECA, the Companies will prepare a release assessment report to place in perspective ~~PFOA is reported for the exhaust gas bubbler aqueous solution at a concentration above the LOQ for the three runs for a given test substance composite, then~~ the potential for PFOA release from full-scale municipal and/or medical waste incineration, as applicable, (including application of air pollution controls) ~~of the subject material~~ in the United States ~~will be assessed to put the data into perspective~~. At a minimum, the report will follow the general outline described below and will state assumptions, document the basis for the assumptions made, quantitatively estimate the variability of calculated estimates (based on the variability of the parameters in the evaluation), and qualitatively discuss the uncertainty of calculated estimates.

1.0 Introduction

- Statement of objective for combustion testing of test substance composites.
- Applicability of the laboratory-scale combustion testing to full-scale municipal waste combustors (MWCs) and/or medical waste incinerators (as applicable) in the United States.

2.0 Summary of study results

- A listing of exhaust gas analytical results reported for each applicable test substance composite.
- A listing of test substance composite analytical results reported for each applicable test substance composite.

3.0 Discussion

- Description of the combustion section of the applicable waste incineration process(es) being evaluated (MWC and/or medical waste incinerator) including the rationale for selecting test target temperature(s) and description of typical operational parameters. Cross-reference to or submission of relevant parts of Appendix D.4 of this ECA can satisfy this provision.

- Description of the post-combustion air pollution control equipment (e.g., lime scrubbing, carbon adsorption) employed by typical operating full-scale waste incineration process(es) as applicable.

4.0 Extrapolation of laboratory test results to the typical waste incineration process(es), as applicable, described in Section 3.0 (above) for each test substance composite to be evaluated.

- The relevance of the subject test substance composite to MWCs and/or medical waste incinerators.
- The estimated concentration of the subject test substance composite to the applicable type(s) of waste incinerator. Available information on hydrogen fluoride concentration in waste incinerator exhaust can provide the basis for an upper bound on this estimated concentration.
- A description of the extrapolation.
- A description of any assumptions used.
- Any unique qualitative or quantitative descriptors of the test, the testing equipment, and the results deemed necessary for informative review of the test and test results. 5.0 Sensitivity Analysis
- Assessment of the impact of variability (quantitative) and uncertainty (qualitative) in each parameter on the evaluation results.

6.0 Conclusions

7.0 References

APPENDIX F

COPY OF EPA ORDER INCORPORATING THIS ENFORCEABLE CONSENT AGREEMENT

APPENDIX F

UNITED STATES
ENVIRONMENTAL PROTECTION AGENCY

TESTING CONSENT ORDER FOR THE LABORATORY-SCALE INCINERATION TESTING OF TELOMERS

Docket No. OPPT - 2004 - 0001

Under the authority of section 4 of the Toxic Substances Control Act (TSCA), 15 U.S.C. 2603, the United States Environmental Protection Agency (EPA) issues this testing consent order (Order) to take effect on the date of publication of the notice in the Federal Register announcing the issuance of this Order. This Order incorporates the enforceable consent agreement (ECA) for the laboratory-scale incineration testing of fluorotelomer based polymer test substance composites listed in Appendix A of the ECA.

____
Date

____
Stephen L. Johnson,
Assistant Administrator
for Prevention, Pesticides,
and Toxic Substances

APPENDIX F

UNITED STATES
ENVIRONMENTAL PROTECTION AGENCY

TESTING CONSENT ORDER FOR THE LABORATORY-SCALE INCINERATION TESTING OF FLUOROPOLYMERS

Docket No. OPPT - 2003 - 0071

Under the authority of section 4 of the Toxic Substances Control Act (TSCA), 15 U.S.C. 2603, the United States Environmental Protection Agency (EPA) issues this testing consent order (Order) to take effect on the date of publication of the notice in the Federal Register announcing the issuance of this Order. This Order incorporates the enforceable consent agreement (ECA) for the laboratory-scale incineration testing of the fluoropolymer test substance composites listed in Appendix A of the ECA.

Date

Stephen L. Johnson,
Assistant Administrator
for Prevention, Pesticides,
and Toxic Substances

APPENDIX G

QUALITY ASSURANCE PLAN OUTLINE

[NOTE: Drafting Committee discussions are continuing to finalize this section]

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