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Oppt-2003-0071-0041



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12/30/03 08:30 PM

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cc: John Blouin/DC/USEPA/US@EPA, Greg Fritz/DC/USEPA/US@EPA  
Subject: Jan 6 Fluoropolymer Incineration ECA development meeting

The next Fluoropolymer Incineration ECA development teleconference is scheduled for January 6th. Attached is Draft #4 of the ECA document which incorporates the changes we discussed during the December 22 teleconference. Revised Appendices were not available in time to distribute with this e-mail. If we don't receive something from Robert Giraud before the conference call then we will use the 10-21-03 version for our discussions (electronic copy attached).

**TENTATIVE AGENDA**

- 1) Introductions
- 2) Status of ECA development
- 3) Continued discussions of ECA Draft #4
- 4) ECA Appendices development
- 5) Other discussions (to be determined by the group)
- 6) Next Steps

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**ATTACHMENTS**

1) ECA Draft #4



ECA\_FluoroIncin\_dft\_1\_6\_04.PC

2) 10/21/03 Giraud Appendices



FP Incin Test Program draft 10-21-03.

\*\*\*\*\*

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**DRAFT #4 TO PFOA ECA PROCESS DRAFTING COMMITTEE**

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

**Docket No. OPPT - [YEAR] - [EDOCKET NO.]**

**[DRAFT 1/6/04]  
[Month Year]**

**NOTE TO DRAFTING COMMITTEE:**

This 1/6/04 draft incorporates agreed changes as discussed at the 12/22/03 meeting. Red text indicates places where suggested revisions have been inserted from the last discussions. Strikeout shows text to be deleted. Green text is used either to show proposed alternative language or to show a series of text still under discussion. Areas for **Follow-on DISCUSSION** usually include: 1) a brief summary in *italics*, 2) original text, 3) FMG proposed text, and in some cases 4) proposed alternative text.

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

Docket No. [OPPT-?YEAR-EDOCKET NO. XXXX?]

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~~H. Interim Progress Report Outline~~

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1 **I. INTRODUCTION**

2  
3 Under the authority of section 4 of the Toxic Substances Control Act (TSCA), 15 U.S.C.  
4 2603, and 40 CFR Part 790 of the Agency's implementing regulations, the United States  
5 Environmental Protection Agency (EPA) and Asahi Glass Fluoropolymers USA, Inc., Daikin America,  
6 Inc., Dyneon, LLC, and E.I. du Pont de Nemours and Company (hereinafter collectively "the  
7 Companies") enter into this enforceable consent agreement (ECA). This ECA will take effect on the  
8 date of publication of the notice in the Federal Register announcing the issuance of the testing consent  
9 order (Order) that incorporates this ECA.

10  
11 On April 16, 2003, EPA initiated a public process to negotiate enforceable consent agreements  
12 (ECAs) under section 4 of TSCA concerning perfluorooctanoic acid (PFOA) and fluorinated telomers  
13 to develop environmental fate and transport information, as well as relevant information to enhance  
14 understanding of the sources of PFOA in the environment and the pathways by which human exposure  
15 to PFOA is occurring (68 FR 18626; April 16, 2003). The goal of the ECAs resulting from these  
16 public discussions is to develop data relevant to identifying the pathway or pathways that result in  
17 exposures to PFOA by air, water, soil, or food; and to characterize how PFOA gets into those  
18 pathways (including the products or processes that are responsible for the presence of PFOA in the  
19 environment). EPA anticipates that the data to be developed under such ECAs will be beyond or  
20 supplemental to that of ongoing testing efforts described under industry letters of intent (LOIs) (Refs 1-  
21 4). [OPPT-2003-0012-0007,0012,0013,0016]

22  
23 In preparation for the June 6, 2003, public meeting, EPA developed a preliminary framework  
24 document outlining data needs that the Agency deemed appropriate to address the outstanding PFOA  
25 source and exposure questions identified in the *Federal Register* notice of April 16, 2003 (Ref  
26 5)[OPPT-2003-0012-0056]. The intent of EPA's preliminary framework document was to serve as a  
27 discussion guide for the June 6, 2003, public meeting and to aid in distinguishing between outstanding  
28 EPA data needs and industry LOI commitments. The preliminary framework document was not a  
29 predetermined list of information needs defining the outcome of the ECA process.

30  
31 This ECA provides for a laboratory-scale incineration testing program of fluoropolymers, which  
32 is one of the data needs identified in EPA's preliminary framework document for PFOA. On June 6,  
33 2003, the PFOA Plenary Group (consisting of EPA and all interested parties) acknowledged such a  
34 testing program as an opportunity for ECA development and tasked the Fluoropolymer Technical  
35 Workgroup to work out the details that could be incorporated into an ECA between test sponsors and  
36 EPA. On July 9, 2003, the Fluoropolymer Technical Workgroup received proposals from the  
37 Companies and EPA for incineration testing of fluoropolymers. Details of this testing program were



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1 developed by members of the Fluoropolymer Incineration Subgroup of the Fluoropolymer Technical  
2 Workgroup during subsequent meetings. On [Month/Date], 2003, the Fluoropolymer Technical  
3 Workgroup acknowledged that this testing program had sufficient merit for consideration by the Plenary  
4 Group. On [Month/Date], 2003, the Plenary Group discussed the merit of this testing program and  
5 recommended that EPA consider entering into an ECA with test sponsors. The official record for the  
6 development of this ECA, including the public version, is established under EPA docket control number  
7 [OPPT-2003-0012]. The procedures for ECA negotiations are described at 40 CFR 790.22(b). The  
8 official record for the testing conducted under this ECA is Docket No. [OPPT-?YEAR-EDOCKET  
9 NO. XXXX?]

10  
11  
12 **II. TEST SUBSTANCES**

13  
14 The chemicals subject to this ECA are the fluoropolymers listed in Appendix A.1.<sup>1</sup> For the  
15 purpose of testing under this ECA the chemicals listed in Appendix A.1 will be combined to form four  
16 composites (see Appendix A.3 and A.4). These composites are representative of fluoropolymer  
17 products manufactured by the Companies that are currently available in the marketplace. The  
18 Companies will provide the fluoropolymers specified in Appendix A.1 for incorporation into the  
19 composites that will be tested under this ECA.<sup>2</sup> Criteria for the selection of each composite to be  
20 tested under this ECA are described in Appendix A.2 of this ECA<sup>1</sup>. The four composites to be tested  
21 are defined for purposes of this ECA as:

- 22  
23 (A) Dry Non-Melt PTFE Resin Composite: Ethene, tetrafluoro-,  
24 homopolymer, CAS No. 9002-84-0,  
25  
26 (B) Dry Melt Fluoropolymer Resin Composite: (containing: 1-Propene,  
27 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No.  
28 25067-11-2; Propane Propene, 1,1,1,2,2,3,3-heptafluoro-3-  
29 [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No.  
30 26655-00-5; Ethene, tetrafluoro-, polymer with

---

<sup>1</sup> There is a Public and CBI version of Appendices A.1, ~~A.2, A.3, and A.4~~ [To be determined] 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).

<sup>2</sup> See the Tables in Part XXIII. of this ECA for ~~list of chemicals subject to each of the Companies the chemicals to be supplied by each Company.~~

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1 trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; 1-Propene,  
2 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and  
3 tetrafluoroethene, CAS No. 25190-89-0; 1-Hexene,  
4 3,3,4,4,5,5,6,6,6-nonafluoro-, polymer with ethene and  
5 tetrafluoroethene, CAS No. 68258-85-5; and, 1-Propene,  
6 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene,  
7 CAS No. 35560-16-8),  
8

9 (C) Dry Non-Melt Fluoroelastomer Gum Composite: (containing: 1-  
10 Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene,  
11 CAS No. 9011-17-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer  
12 with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0;  
13 1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene,  
14 CAS No. 54675-89-7; 1-Propene, polymer with tetrafluoroethene,  
15 CAS No. 27029-05-6; Ethene, tetrafluoro-, polymer with  
16 trifluoro(trifluoromethoxy) ethene, CAS No. 26425-79-6; and, Ethene,  
17 chlorotrifluoro-, polymer with 1,1-difluoroethene, CAS No. 9010-75-  
18 7; and ??generic name??. **Accession No. ??????**, and  
19

20 (D) Aqueous Fluoropolymer Dispersions Composite: (containing: Ethene,  
21 tetrafluoro-, polymer with trifluoro(pentafluoroethoxy) ethene, CAS  
22 No. 31784-04-0; Ethene, tetrafluoro-, homopolymer, CAS No.  
23 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with  
24 tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-  
25 heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene,  
26 CAS No. 26655-00-5; Ethene, tetrafluoro-, polymer with  
27 trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; and 1-  
28 Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and  
29 tetrafluoroethene, CAS No. 25190-89-0.  
30

31 The procedure for constructing each composite is described in Appendix A.4 to this ECA<sup>1</sup>.  
32 ~~Components of each fluoropolymer composite to be tested must be as pure as can be reasonably~~  
33 ~~attained. ,and in any event must not be less than XX percent pure.~~ The polymer components for each  
34 ~~composite~~ will be unfilled first quality product polymer, substantially free of inorganic constituents. Each  
35 component of the four composites to be tested under this ECA will be accompanied by a certificate of  
36 analysis ,and will be shown showing it to meet applicable product specifications.  
37

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1 **III. OBLIGATION OF SIGNATORY COMPANIES**

2  
3 A. The Companies are bound by the terms of this ECA as specified below.

4  
5 B. Each Company shall be responsible for supplying the test substance(s) it manufacturers  
6 for incorporation into the composite(s) to be tested under this ECA, as specified on each Company  
7 signature page and in Appendix A.3. The schedule for the testing program includes the deadline date  
8 by which the Companies must submit their contribution(s) to the facility(ies) that will be assembling the  
9 composites to be tested under this ECA. Any Company failing to comply with this ECA requirement  
10 will be in violation of this ECA as described in 40 CFR 790.65 (see part XI of this ECA). In the event  
11 that one or more of the Companies are in violation as described above then the remaining Companies  
12 will inform EPA of the problem and request an EPA determination on how to proceed with the testing  
13 program described under this ECA. Each Company required to contribute to a particular composite is  
14 obligated to complete the testing required by this ECA for that composite. A Company shall not be  
15 responsible for any failure to perform its obligation under this ECA that is caused by circumstances  
16 beyond their control, that the Company could not have prevented through the exercise of due diligence.  
17 Under such circumstances the Company will consult with EPA to ~~determine reach agreement on~~ what  
18 modifications, if any, are needed in the test plan or scope of testing (see Part X of this ECA regarding  
19 modification to this ECA as contained in 40 CFR 790.68).

20  
21 C. The Companies recognize that to implement this ECA, EPA will issue an Order under  
22 section 4 of TSCA that incorporates the terms of this ECA (see Appendix G). The Companies agree  
23 that all terms of this ECA will take effect on the date of publication of the notice in the Federal Register  
24 announcing the issuance of the Order that incorporates this ECA, and all applicable all time periods that  
25 will be treated as beginning on that publication date.

26  
27  
28 **IV. PRINCIPAL TEST SPONSOR**

29  
30 The Companies ~~are members of have identified~~ the Fluoropolymer Manufacturers Group  
31 (FMG), ~~which represents the manufacturers of the fluoropolymer chemicals subject to this ECA as~~  
32 ~~described in Part II and listed in Appendix A.1. In accordance with 40 CFR 790.60 (4) and 40 CFR~~  
33 ~~790.65 (d) the Companies, in their sole discretion, may elect to use FMG to administer the test~~  
34 ~~program under this ECA, and to communicate with EPA about schedules, study plans, protocols, test~~  
35 ~~standards, and other aspects of the testing program. In performing these functions, the CTF will be~~  
36 ~~acting as the agent of the Companies for purposes of communication with EPA. EPA and the~~  
37 ~~Companies acknowledge that, the CTF agree that FMG has no legal responsibility for complying with~~  
38 this ECA. Responsibility for complying with the ECA rests at all times with the Companies.

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1  
2 **V. PURPOSE OF THE TESTING PROGRAM**  
3

4           The purpose of the testing program specified by this ECA is to assess the potential for waste  
5 incineration of fluoropolymers (see Part II and Appendix A.1 of this ECA) to emit PFOA, based on  
6 quantitative determination of potential exhaust gas levels of PFOA that may emanate from laboratory-  
7 scale combustion testing under conditions representative of typical municipal waste combustor  
8 operations in the United States.  
9

10           EPA believes that these incineration studies of fluoropolymers will develop data needed by the  
11 Agency to determine whether municipal and/or medical waste incineration of fluoropolymers is a  
12 potential source of PFOA that may contribute as a pathway to environmental and human exposures.  
13 The data may also be used to inform screening level human and environmental exposure assessments.  
14 In addition, the data may also be used by other Federal agencies (e.g., the Agency for Toxic  
15 Substances and Disease Registry (ATSDR), the National Institute for Occupational Safety and Health  
16 (NIOSH), the Occupational Safety and Health Administration (OSHA), and the Consumer Product  
17 Safety Commission (CPSC), the Food and Drug Administration (FDA)) in assessing chemical risks and  
18 in taking appropriate actions within their programs. It is intended that the data generated under this  
19 ECA will identify whether the incineration of fluoropolymers contributes to the sources and pathways of  
20 environmental and human exposure to PFOA.  
21  
22

23 **VI. SCOPE OF THE PROGRAM**  
24

25           This testing program will consist of the testing listed in Table 1 in accordance with the test  
26 standards specified in Table 1 and described in Appendices B.?? and C1 - C2.6 as annotated by  
27 Appendix D.?? to this ECA ("Test Standards") and submitting the reports and documents specified in  
28 Table 1 in accordance with the deadlines set forth in Table 1 and described in Appendices C.1 - C.2.6  
29 and E.  
30

31  
32 **VII. DESCRIPTION OF THE TESTING PROGRAM**  
33

34           The program has two segments as follows: Phase I PFOA Transport Testing and Phase II  
35 Fluoropolymer Incineration Testing.  
36

37           A.    Phase I PFOA Transport Testing: Phase I will consist of quantitative transport  
38 efficiency testing for PFOA. Phase I testing for PFOA transport efficiency is specified in the Phase I

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

8  
9 **B. Phase II Fluoropolymer Incineration Testing:** This testing, specified in the Phase II  
10 Fluoropolymer Incineration Testing segment of Table 1 and described in Appendix C2.1 - C2.6 as  
11 annotated by Appendices B.?? and D.?? will include the following for each fluoropolymer composite to  
12 be tested under this ECA: 1) elemental analysis, 2) combustion stoichiometry, 3) thermogravimetric  
13 analysis, 4) laboratory-scale combustion testing, and, 5) if required under this ECA,<sup>3</sup> release  
14 assessment reporting.

15  
16  
17 **VIII. PHASE I TECHNICAL CONSULTATION**

18  
19 **A.** Following completion of Phase I and prior to the initiation of Phase II, the Companies will  
20 submit a letter report to EPA with the results for the recovery across the laboratory-scale thermal  
21 reactor system, as determined from Phase I testing.

22  
23 **B.** If the recovery for either PFOA or Total Fluorine (as determined by the formulas in  
24 Appendix C.1) is greater than or equal to 70%, the Companies will proceed to Phase II testing.

25  
26 **C.** If the recovery for both PFOA and Total Fluorine (as determined by the formulas in  
27 Appendix C.1) is less than 70%, a Technical consultation will be held. The objective of the Technical  
28 Consultation will be to reach agreement on how to proceed. Specifically, the technical consultation will  
29 address: (1) whether the data from the Phase I PFOA Transport Testing segment provide a sufficient  
30 basis for conducting the laboratory-scale incineration testing specified in the Phase II Fluoropolymer

---

<sup>3</sup> 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.3) 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 applicable full-scale ~~municipal and/or medical~~ waste incinerator operations in the United States.

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1 Incineration Testing segment; (2) the nature and scope of any additional Phase I work that may be  
2 required prior to the commencement of Phase II Testing and reporting (e.g., modifications to the  
3 Advanced Thermal Reactor System) as described in Part VII. B. of this ECA), and/or (3) the nature  
4 and scope of modifications to the protocols and test standards for Phase I and/or Phase II testing that  
5 may be needed to complete the testing under this ECA. The technical consultation will review the  
6 outcomes of the Phase I PFOA Transport Efficiency Testing, to discuss the feasibility of proceeding  
7 with Phase II Testing as described in this ECA, and to ~~determine~~ discuss whether additional  
8 modifications are needed to the test standards and/or protocols described in Appendices B, C and D  
9 for Phase I PFOA Transport Testing and/or Phase II Fluoropolymer Incineration Testing.

10  
11 Possible outcomes of the Technical Consultation include, but are not limited to, the following:

- 12 1. An agreement to conduct additional Phase I testing,  
13 and the schedule and standards for such testing, to  
14 inform whether and under what conditions to conduct  
15 Phase II testing.
- 16 2. An agreement to proceed into Phase II testing with or  
17 without agreed-to modifications to plans, test standards  
18 and schedules for Phase II testing.
- 19 3. No agreement on a path forward, in which case the  
20 Companies' obligations to conduct testing or reporting  
21 beyond Phase I PFOA Transport Testing as described  
22 in this ECA are terminated.  
23  
24  
25  
26

27 D. EPA shall place in the record for this action a summary of any Technical Consultation  
28 meeting that is held under this paragraph. In the event modifications to the scope of the testing program  
29 are ~~deemed necessary~~ agreed to, EPA and the Companies will revise this ECA, as well as Table 1 and  
30 the Appendices, as appropriate. If EPA determines that any changes to the scope of the testing  
31 program are significant, an opportunity for public participation will be provided. EPA will publish a  
32 request for public comments and/or announce a public meeting in the Federal Register. Following the  
33 comment period, EPA will place in the record for this action a summary of the Technical Consultation,  
34 a copy of comments received, a copy of the letter sent to the Companies explaining the decisions and  
35 outcomes of the technical consultation, and a copy of the modified ECA. This process is in lieu of the  
36 modification procedures described in 40 CFR 790.68 (see Part X of this ECA).  
37  
38

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1 **IX. STANDARDS FOR CONDUCTING TESTING**

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

11  
12 Provisions that are not mandatory, and are therefore only recommended, are identified by the  
13 use of "should" statements. In the event such "should" provisions are not followed, the Companies will  
14 not be deemed by EPA to be in violation of this ECA and will not be subject to penalties or other  
15 enforcement actions, as described in Part XI. of this ECA. However, in such cases, EPA will use its  
16 professional judgement to determine the scientific adequacy of the test results and any repeat testing  
17 that is determined by EPA to be necessary will be required either under a separate ECA or pursuant to  
18 a rule promulgated under section 4(a) of TSCA, 15 U.S.C. 2603(a).

19  
20 B. The Companies and EPA will consult in a good faith effort to consider the need for Test  
21 Standard modifications if either EPA or the Companies desire such modifications. Modifications to this  
22 ECA will be governed by 40 CFR 790.68 (see Part X. of this ECA).

23  
24 **\*\*\* Follow-on DISCUSSION POINT:**

25 [For a future meeting: The FMG will provide a table demonstrating how the QAPjP will  
26 address the GLP requirements under 40 CFR part 792.]

27  
28 {12/22/03 Revised placeholder text with additional 1/6/04 text to be discussed}

29 C. All testing required by this ECA must be conducted in accordance with the  
30 EPA Good Laboratory Practice Standards (GLPS) found at 40 CFR part 792, except ~~as~~  
31 ~~provided in Appendix F~~ as follows ~~.....~~ (list to be developed from FMG table).

32  
33 {11/24/03 FMG proposed revisions}

34 C. All testing required by this ECA must be conducted in accordance with ~~the EPA Good~~  
35 ~~Laboratory Practice Standards (GLPS) found at 40 CFR part 792.~~ a Quality Assurance Project  
36 Plan prepared in accordance with Appendix YYY.<sup>3</sup>

37  
38 {original text}

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1 C. All testing required by this ECA must be conducted in accordance with the EPA Good  
2 Laboratory Practice Standards (GLPS) found at 40 CFR part 792.  
3  
4

5 **X. STUDY PLANS**  
6

7 The Companies will submit a study plan to EPA for each test conducted pursuant to this ECA  
8 prior to the initiation of testing in accordance with 40 CFR 790.62. (For this ECA, EPA will not  
9 require the plans under this Part of the ECA to be submitted "no later than 45 days prior to the initiation  
10 of testing," as specified at 40 CFR 790.62(a)). The content of the study plan plans submitted to EPA  
11 will comply with 40 CFR 790.62(b). The A study plan may cross reference those specific portions of  
12 Table 1 and/or the Appendices. Also pursuant to Part IX. (C) of this ECA, the Companies must  
13 submit a Quality Assurance Project Plan Plans (QAPjP) prepared in accordance with Appendix F  
14 EPA guidance.<sup>4</sup> Modifications to the study plans under this part of the ECA will be governed by the  
15 procedures of 40 CFR 790.62(c) except that the 15 day time periods in 40 CFR 790.62(c) (2) and (3)  
16 will be 45 day time periods.. All study plans will become part of the official record (Docket Control  
17 Number [OPPT-YEAR-EDOCKET NO. ?? XXXX ??]).  
18  
19

20 **XI. MODIFICATIONS TO THIS ENFORCEABLE CONSENT AGREEMENT**  
21

22 Except as provided in this agreement, modifications to this ECA, if any, will be made according  
23 to the procedures contained in 40 CFR 790.68.  
24  
25

26 **XII. FAILURE TO COMPLY WITH THE ENFORCEABLE CONSENT AGREEMENT**  
27

28 The Companies acknowledge that a violation of the requirements of this ECA will constitute a  
29 "prohibited act" under section 15(1) of TSCA, 15 U.S.C. 2614(1), and will trigger all provisions  
30 applicable to a section 15 violation. In addition, the Companies acknowledge that noncompliance with  
31 any term of this ECA by any Company will constitute conduct "in violation of this Act" under section  
32 20(a)(1) of TSCA, 15 U.S.C. 2619(a)(1), and could result in a citizen's civil action.  
33

---

<sup>4</sup> Guidance for developing Quality Assurance Project Plans can be found in the EPA document EPA QA/G-5: *Guidance for Quality Assurance Project Plans*, prepared by: Office of Environmental Information, EPA, December 2002. This is also available from the EPA website at <http://epa.GOV/Quality/qs-docs>.



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1 Under the penalty provisions of section 16 of TSCA, 15 U.S.C. 2615, and the Federal Civil  
2 Penalties Inflation Adjustment Act of 1990, 28 U.S.C. 2461 note, as amended by the Debt Collection  
3 Improvement Act of 1996, 31 U.S.C. 3701 note, as implemented by 61 FR 69360 (December 31,  
4 1996), a non-complying Company could be subject to a civil penalty of up to \$27,500 per violation,  
5 with each day in violation potentially constituting a separate violation under section 15. Knowing or  
6 willful violations may lead to the imposition of criminal penalties, or a fine of not more than \$27,500 for  
7 each day of violation, or imprisonment for not more than one year, or both. In addition, EPA could  
8 enforce this ECA pursuant to section 17 of TSCA, 15 U.S.C. 2616, by seeking an injunction to  
9 compel adherence to the requirements of this ECA.

10  
11  
12 **XIII. EPA MONITORING OF ENFORCEABLE CONSENT AGREEMENT TESTING**

13  
14 EPA may conduct monitoring activities of the testing conducted under this ECA such as  
15 laboratory inspections and study audits, as permitted under section 11 of TSCA, 15 U.S.C. 2610.

16  
17  
18 **XIV. SUBMISSIONS TO EPA AND CONFIDENTIALITY OF INFORMATION**

19 **\*\*\* Follow-on DISCUSSION POINT:** *[Summary - EPA considered the 12/10 FMG*  
20 *proposed language and provided clarification text on 12/22/03. FMG rejected EPAs*  
21 *clarification and asked EPA to reconsider. As of 12/30/03 EPA continues internal deliberation*  
22 *on this point]*

23  
24 **{FMG proposed 12/10/03 additional text insert}**

25 A paper copy of a document shall be deemed submitted when it is either postmaked or placed  
26 in the hands of a commercial courier service for delivery to EPA at the appropriate address specified  
27 above. Hand-delivered documents are deemed submitted upon receipt. Electronically transmitted  
28 documents are deemed delivered upon transmission.

29  
30  
31 **{Text proposed by EPA in 12/22/03 draft}**

32 A. All final reports must be submitted by the Companies to EPA by the dates specified in  
33 Table 1 unless otherwise authorized by EPA pursuant to 40 CFR 790.68. A report will be deemed  
34 submitted when it is date stamped on the day it is received in the Agency's Office of Pollution  
35 Prevention and Toxics (OPPT) Document Control Office (see part XIII B. of this ECA).

36  
37 In accordance with 40 CFR 790.62 (d), the Companies, through the principal test sponsor, will  
38 submit interim progress reports to EPA informing the Agency of any proposed changes in standards for

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1 the development of data, study plans, or test schedules, and communicating with the Agency about  
2 laboratory inspections and other matters affecting the progress of testing. The schedule for interim  
3 progress reports is specified in Table 1 of this ECA. A suggested outline for interim progress reporting  
4 is provided in Appendix E.2. **[Note to Drafting Committee: Please help me remember... I**  
5 **believe that we agreed to accepting this paragraph although we have not as yet discussed the**  
6 **draft Appendix E.2]**

7  
8 B. All documents submitted to EPA under this ECA must be identified by the Docket ID  
9 Number **(OPPT- Year - EDOCKET NO. ?? XXXX ??)** and the name: ECA on Laboratory-Scale  
10 Incineration Testing of Fluoropolymers. Submissions made by mail should be sent to: Document  
11 Control Office (7407M), Office of Pollution Prevention and Toxics (OPPT), Environmental Protection  
12 Agency, 1200 Pennsylvania Avenue, NW, Washington, DC 20460-0001. Submissions made by hand  
13 delivery or courier should be delivered to: OPPT Document Control Office (DCO) in the EPA East  
14 Building, Room 6428, 1201 Constitution Avenue, NW, Washington, DC and marked Attention:  
15 Docket ID Number **OPPT- Year - ??XXXX??**. The DCO is open from 8 a.m. to 4 p.m., Monday  
16 through Friday, excluding legal holidays. The telephone number for the DCO is (202) 564-8930.

17  
18 C. The Companies must submit six (6) paper copies of each version (Public and CBI) for  
19 all reports described in Table 1 and Part VI A. and B. of this ECA. In addition, an electronic file of all  
20 documents submitted under this ECA (marked as CBI where appropriate and in text-searchable, PDF  
21 format) will be provided to EPA.

22  
23 D. Any document submitted to EPA that contains data or information for which a  
24 Signatory Company makes a claim of confidentiality (see Part XV of this ECA), must be submitted as  
25 two separate versions. One version must be complete, with the information being claimed as  
26 confidential marked in the manner described under 40 CFR 790.7. The other, public version must be  
27 identical in all respects except that all of the information claimed as confidential shall be redacted. EPA  
28 will place the public version in the Agency's docket. The complete version will be treated in  
29 accordance with EPA confidentiality regulations in 40 CFR part 2 and 40 CFR 790.7.

30  
31 Data or other information that are considered to be CBI must not be submitted through EPA's  
32 electronic public docket or by e-mail. Any part or all of data or other information claimed as CBI must  
33 be so marked. If the CBI submission is on diskette or CD ROM, mark the outside of the diskette or  
34 CD ROM as CBI and then identify electronically within the diskette or CD ROM the specific  
35 information that is CBI. Information marked as CBI will not be disclosed except in accordance with  
36 procedures set forth in 40 CFR part 2 (see Part XV of this ECA).

37

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

7  
8  
9 **XV. PUBLICATION AND DISCLOSURE OF TEST RESULTS**

10 **\*\*\* Follow-on DISCUSSION POINT** *[Summary - FMG proposed 11/24/03 additional text to*  
11 *clarify conditions under which EPA can share a CBI Document with another government*  
12 *agency. EPA struck this addition on 12/22/03 citing laws governing such distribution adequately*  
13 *addressed FMG concerns. During further discussion, it became clear that additional*  
14 *clarification could be provided to meet FMG's needs. Alternative language is suggested in*  
15 *green.]*

16  
17 All results of testing conducted pursuant to this ECA will be announced to the public by EPA in  
18 accordance with the procedures specified in section 4(d) of TSCA, 15 U.S.C. 2603(d). Disclosure  
19 by EPA of data generated by such testing to the public or other government agencies will be governed  
20 by section 14(b) of TSCA, 15 U.S.C. 2613(b), and 40 CFR part 2. The CBI version of a document  
21 will only be provided to another U.S. government organization in compliance with the procedures  
22 described in the OPPTS TSCA CBI Procedure Manual.

23  
24 **{FMG 11/24/03 proposed additional text / struck by EPA 12/22/03}**

25 ~~The CBI version of a document will not be provided to another government agency unless that agency~~  
26 ~~has certified that it affords equivalent protection.~~

27  
28  
29  
30 **XVI. OTHER RESPONSIBILITIES OF THE COMPANIES**

31  
32 **\*\*\* Follow-on DISCUSSION POINT** *[Summary - As of 12/30/03 EPA continues internal*  
33 *deliberation on the FMG request to modify Part XVI A. of this ECA]*

34  
35 A. The Companies will comply with the notification requirements of section 12(b)(1) of  
36 TSCA, 15 U.S.C. 2611(b)(1), and 40 CFR part 707, subpart D, if they export or intend to export any  
37 of the fluoropolymer chemicals listed in Appendix A.1 to this ECA. Any other person who exports or

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1 intends to export any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA is subject to  
2 the above cited export notification requirements

3  
4 B. If any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA become  
5 subject to a rule promulgated under TSCA section 5(a)(2), 15 U.S.C. 2604(a)(2), governing significant  
6 new uses of any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA, then the  
7 Companies will be subject to the data submission requirements imposed by section 5(b)(1)(A) of  
8 TSCA, 15 U.S.C. 2604(b)(1)(A), as if the testing under this ECA had been required by a TSCA  
9 section 4 test rule.

10  
11  
12 **XVII. SEVERABILITY OF ENFORCEABLE CONSENT AGREEMENT PROVISIONS**

13  
14 In the event that one or more provisions of this ECA are determined by a court decision to be  
15 unenforceable, the remaining provisions of this ECA will not be presumed to be valid, and EPA will  
16 either initiate a rulemaking proceeding to require testing or publish in the Federal Register the reasons  
17 for not initiating such a proceeding.

18  
19  
20 **XVIII. FINAL AGENCY ACTION**

21  
22 For purposes of 5 U.S.C. 704, publication of the FR notice announcing the issuance of the  
23 Order incorporating this ECA constitutes final agency action..

24  
25  
26 **XIX. PUBLIC RECORD**

27  
28 EPA has established a public record which will contain this ECA, the Order that incorporates  
29 this ECA, the Federal Register notice announcing issuance of the Order incorporating this ECA, and  
30 any and all relevant information, subject to the confidentiality provisions of section 14(b) of TSCA and  
31 40 CFR part 2. The official record for this ECA, including the public version, which does not include  
32 any information claimed as CBI, has been established under Docket Control Number [OPPT-YEAR-  
33 **EDOCKET NO. ?? XXXX ??**].

34  
35 An electronic version of the public docket is available through EPA's electronic public docket  
36 system, EPA Dockets. EPA Dockets may be accessed at <http://www.epa.gov/edocket/> to access the  
37 index listing of the contents of the official public docket, and to access those documents in the public  
38 docket that are available electronically. Although not all docket materials may be available

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1 electronically, (for example the materials in the original dockets for this action, [AR-226 and OPPTS-  
2 2003-0012], or materials under copyright), can be access any of the publicly available docket materials  
3 through the EPA Docket Center, Rm. B102-Reading Room, EPA West, 1301 Constitution Ave.,  
4 NW., Washington, DC. For materials available in the electronic docket, once in the system, select  
5 "search," then key in the appropriate docket ID number [(OPPT-YEAR-EDOCKET NO. XXXX).]  
6  
7

8 **XX. EFFECTIVENESS**

9  
10 This ECA may be signed in separate counterparts. This ECA will not be effective unless signed  
11 by each of the Companies and by EPA. This ECA will take effect on the date of publication of the  
12 Federal Register notice announcing the issuance of the Order that incorporates this ECA.  
13  
14

15 **XXI. RIGHTS OF THE COMPANIES**

16  
17 By signing this ECA, the Companies waive their right to challenge EPA's authority to assess  
18 penalties for violations of the terms of this ECA. This waiver does not affect any other rights that the  
19 Companies may have under TSCA, including the right to dispute the amount of any penalty or to  
20 dispute factually whether a violation of the terms of this ECA has occurred, or to seek judicial review of  
21 any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer  
22 chemicals listed in Appendix A.1 to this ECA.  
23

24 **\*\*\* Follow-on DISCUSSION POINT** [Summary - for the 11/24/03 meeting the FMG  
25 proposed additional text to this Part of the ECA. EPA attempted to clarify this language at the  
26 12/22/03 meeting. The EPA clarifying language was rejected by FMG. FMG asserted that they  
27 consider their additional text as RESERVED text for the Companies. As of 12/30/03 EPA  
28 continues internal deliberation to assess this additional language insert.]  
29

30 **{ FMG 11/24/03 additional language}**

31 By signing this ECA, the Companies are not admitting that the requirements of TSCA Section 4  
32 have been satisfied for promulgating a test rule to generate the data required by this ECA.  
33  
34

35 **{ EPA suggested 12/22/03 revision (CAPS/strikeout) to 11/24/03 proposed FMG additional**  
36 **ECA text}**

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1           By signing this ECA, the Companies are not admitting that the ~~requirements of~~ *FINDINGS*  
2 *REQUIRED BY* TSCA Section 4 (a) ~~have been satisfied~~ for promulgating a test rule ~~to generate the~~  
3 ~~data required by this ECA~~ **HAVE BEEN MADE BY EPA.**

4  
5  
6

1  
2 **XXII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR**  
3

4 The Principal Test Sponsor is:

5  
6 Consortium for Incineration Testing (CIT)  
7 **[? Name of technical contact person ?]**  
8 **[? ADDRESS ?]**  
9 **[? Phone Number ?]**

10  
11 The Companies subject to this ECA are:

12  
13  
14  
15 Asahi Glass Fluoropolymers USA, Inc.  
16 **[? ADDRESS ?]**

17  
18  
19 Daikin America, Inc.  
20 **[? ADDRESS ?]**

21  
22  
23 Dyneon, LLC  
24 **[? ADDRESS ?]**

25  
26  
27 E.I. du Pont de Nemours and Company  
28 **[? ADDRESS ?]**

29

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1 Special Page Header: ECA Copy # 3 Asahi Glass Fluoropolymers USA, Inc.  
2

3 **XXIII. SIGNATURE**

4 **TEST SPONSOR**

5  
6 **Asahi Glass Fluoropolymers USA, Inc.<sup>1</sup>**  
7

8

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

9  
10  
11  
12  
13  
14  
15

16  
17 Company technical contact person for handling correspondence marked as "Confidential"  
18

19 Name: \_\_\_\_\_  
20 Title: \_\_\_\_\_  
21 Address: \_\_\_\_\_  
22 Phone Number: \_\_\_\_\_  
23  
24  
25

26  
27 Date: \_\_\_\_\_  
28 [? NAME ?]  
29 [? TITLE ? e.g., Senior Vice President]

---

<sup>1</sup> Data in the table listing the ECA subject chemicals for Asahi Glass Fluoropolymers USA, Inc. was provide to EPA by the Company. 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 XV of this ECA regarding confidentiality of information).



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1  
2

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

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FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1 Special Page Header: ECA Copy # 4

Daikin America, Inc.

2  
3 **XXIII. SIGNATURE**

4 **TEST SPONSOR**

5  
6 **Daikin America, Inc.<sup>1</sup>**

7

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

8  
9  
10  
11  
12  
13  
14  
15

16  
17 Company technical contact person for handling correspondence marked as "Confidential"

18  
19 Name: \_\_\_\_\_  
20 Title: \_\_\_\_\_  
21 Address: \_\_\_\_\_  
22 Phone Number: \_\_\_\_\_  
23  
24  
25  
26

27 Date: \_\_\_\_\_  
28 **[? NAME ?]**  
29 **[? TITLE ? e.g., Senior Vice President ]**

---

<sup>1</sup> Data in the table listing the ECA subject chemicals for Daikin America, Inc. was provide to EPA by the Company. 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 XV of this ECA regarding confidentiality of information).

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1  
2

Daikin America, Inc.  
[? ADDRESS ?]

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1 Special Page Header: ECA Copy # 5 Dyneon, LLC

2  
 3 **XXIII. SIGNATURE**

4 **TEST SPONSOR**

5  
 6 **Dyneon, LLC<sup>1</sup>**

7

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

8  
 9  
 10  
 11  
 12  
 13  
 14  
 15  
 16  
 17 Company technical contact person for handling correspondence marked as "Confidential"

18  
 19 Name: \_\_\_\_\_  
 20 Title: \_\_\_\_\_  
 21 Address: \_\_\_\_\_  
 22 Phone Number: \_\_\_\_\_

23  
 24  
 25  
 26  
 27 Date: \_\_\_\_\_  
 28 **[? NAME ?]**  
 29 **[? TITLE ? e.g., Senior Vice President ]**

---

<sup>1</sup> Data in the table listing the ECA subject chemicals for Dyneon, LLC was provide to EPA by the Company. 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 XV of this ECA regarding confidentiality of information) .

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1  
2

Dyneon, LLC  
[? ADDRESS ?]

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FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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

**XXIII. SIGNATURE**

**TEST SPONSOR**

**E.I. du Pont de Nemours and Company<sup>1</sup>**

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 ]

---

<sup>1</sup> Data in the table listing the ECA subject chemicals for E. I. du Pont de Nemours and Company was provide to EPA by the Company. 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 XV of this ECA regarding confidentiality of information) .

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**FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1  
2

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

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FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1 Special Page Header:

2 ECA Copy # 1 EPA CONTAINS CONFIDENTIAL BUSINESS INFORMATION

3  
4 Special Page Header:

5 ECA Copy # 2 EPA PUBLIC VERSION  
6 CONTAINS NO CONFIDENTIAL BUSINESS INFORMATION

7  
8  
9 **XXIV. SIGNATURE**

10  
11  
12  
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14  
15  
16  
17 **UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**

18  
19  
20  
21  
22  
23  
24  
25  
26  
27  
28  
29 Date: \_\_\_\_\_

\_\_\_\_\_

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

30  
31  
32  
33  
34  
35 Address:

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



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1

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - December 22, 2003  
 FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1  
2  
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**Table 1 REQUIRED TESTING, TEST STANDARDS, REPORTING AND OTHER REQUIREMENTS FOR THE LABORATORY-SCALE INCINERATION TESTING OF FLUOROPOLYMERS**

<b>Phase I PFOA Transport Testing</b>	<b>Test Standard</b>	<b>Deadline for Final Report (Months)<sup>2</sup></b>
Quantitative PFOA transport analysis <sup>3</sup>	See appendix C.1 as annotated in appendix D.?)	?TBD "4" ? <sup>4</sup>

---

<sup>2</sup> Number of months after the effective date of the Order that incorporates this ECA when final report is due. Interim status reports, describing the status of all testing to be performed under this ECA, must be submitted by the Companies, through the FMG, to EPA every 6 months beginning six months from the effective date of this ECA until the end of the ECA testing program (see Part VIII. D. and Part XIII. of this ECA).

<sup>3</sup> As described in Part VI. A. and B. of this ECA, at the conclusion of Phase I PFOA transport efficiency testing, and prior to initiation of Phase II, the Companies, through the FMG, will provide a letter/report to EPA summarizing the results of Phase I testing. In the event that the transport efficiency of PFOA or of total fluorine (as expressed as the total stoichiometric fluorine content of PFOA and/or as total fluorine) 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 expressed as the total stoichiometric fluorine content of PFOA and/or as total fluorine) is less than 70% then the Companies, through the FMG, 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 VII of this ECA.

<sup>4</sup> In the event that the transport efficiency of PFOA or of total fluorine (as expressed as the total stoichiometric fluorine content of PFOA and/or as total fluorine) is less than 70% and the Technical Consultation concludes that testing can not proceed to Phase II, then the Companies, through the FMG, will submit a complete report for Phase I testing within 60 days following notification of the Technical Consultation outcome. In the event that the outcome of the Technical Consultation indicates that testing can proceed to Phase II Testing then the final report for Phase I will be incorporated into the final report for Phase II Testing.

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - December 22, 2003  
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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<b>Phase II Fluoropolymer Incineration Testing</b>	<b>Test Standard</b>	<b>Deadline for Final Report (Months)<sup>5</sup></b>
Elemental analysis	ASTM D 3176 or equivalent (see Appendix B.? as annotated in Appendix C.2.1)	<u>?TBD "4"?</u>
Combustion stoichiometry	See Appendix C.2.2 as annotated in Appendix D.?	<u>?TBD "4"?</u>
Thermogravimetric analysis	ASTM E 1868-02 (see Appendix B.? as annotated in Appendix C.2.3)	<u>?TBD "6"?</u>
Laboratory-scale combustion testing	Appendix C.2.4 as annotated in Appendix D.?)	<u>?TBD "18"?</u>
Release assessment report <sup>6</sup>	(see Appendix E)	<u>?TBD "20"?</u>

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<sup>5</sup> Number of months after initiation of Phase II testing when final report for this testing is due (see footnotes 2 and 3).

<sup>6</sup> In the event that Phase II Testing identifies measurable levels of PFOA (where measurable PFOA is defined to be at or above the limit of detection (LOD) and, where LOD is identified to be 10 ppt under standard temperature and pressure (see also Appendix D.3)) resulting from the combustion testing for any or all of the fluoropolymer composites to be tested under this ECA, then the Companies, through the FMG, will prepare a release assessment report to put into perspective the relevance of the laboratory-scale incineration testing data with respect to municipal incineration operations in the United States (see Appendix E to this ECA).

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - December 22, 2003  
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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**APPENDIX A**

**TEST SUBSTANCES**

- A.1 List of Chemicals Subject to this ECA**
- 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 CHEMICALS SUBJECT TO THIS ECA<sup>1</sup>

The following table lists the thirteen fluoropolymer chemicals that are the subject to this ECA.

The identities of the fluoropolymers 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 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 -(CF<sub>2</sub>)<sub>x</sub>- which is a potential source of PFOA. For all fluoropolymer products used in commerce, the -(CF<sub>2</sub>)- 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.

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-[[trifluoroethenyl]oxy]-, 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

<sup>1</sup> 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 XV of this ECA regarding confidentiality of information).

PUBLIC VERSION - CONTAINS NO CBI

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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??

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**APPENDIX A.2**

**RATIONALE FOR SELECTING COMPOSITES TO BE TESTED**

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**APPENDIX A.3**

**COMPOSITION OF COMPOSITES TO BE TESTED<sup>1</sup>**

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<sup>1</sup> 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 XV of this ECA regarding confidentiality of information).



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**APPENDIX A.4**

**PREPARATION OF COMPOSITES TO BE TESTED<sup>1</sup>**

---

<sup>1</sup> There is a Public and CBI version of Appendix A.4 because the Companies have asserted that details describing their chemical(s) as a component of the composite(s) is considered by them to be entitled to treatment as TSCA confidential business information (CBI) (see Part XV of this ECA regarding confidentiality of information).

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**APPENDIX B**

**TEST STANDARDS**

- B.1** ASTM E 18680-02 Loss-on-Drying by Thermogravimetry
- B.2** others.....?

\* To be provided by FMG

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**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 Sampling and Analysis**
  - C.2.6 Study Reporting**

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**APPENDIX D**

**ATTACHMENTS AND REFERENCED MATERIALS**

- D.1 Wickbold Torch Method
- D.2 Waste Incineration and Operation Conditions
- D.3 PFOA Analysis Method
- D.4 Other.....

\* To be provided by the FMG

APPENDIX E

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**OUTLINES FOR  
INTERIM STATUS REPORTING  
AND  
RELEASE ASSESSMENT REPORT**

First Draft of Discussion and Comment

APPENDIX E.1

OUTLINE FOR INTERIM REPORTING

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

OPPT Docket ID No: OPPT-Year- XXXXX

Date of Interim Report:

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

1) List of significant ECA Test Program milestones during this six month period:

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

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

1 **APPENDIX E.2**  
2 **Outline for Release Assessment Report**  
3

4 As described in Part VI C., footnote 2, and Table 1 footnote 5 of this document, if the  
5 results of Phase II Fluoropolymer Incineration Testing show that PFOA is quantitated at  
6 greater than the LOD (Limit of Detection), the Companies, through the FMG, will provide a  
7 release assessment report to put the data into perspective relevant to municipal waste  
8 incineration practices in the United States. The objective of this release report is to place the  
9 results of the laboratory-scale incineration test as described in Part VI C. and Table 1 of this  
10 ECA in context with the process of municipal waste incineration in the United States and to  
11 provide sufficient quality information to inform human and environmental exposure  
12 assessments. At a minimum, the report will follow the general outline described below and will  
13 state assumptions, verify the validity of the assumptions made, and evaluate and characterize  
14 the variability and uncertainty of calculated estimates:

15  
16 **1.0 Introduction**

- 17 • Statement of objective for combustion testing of fluoropolymers
- 18 • Applicability of the laboratory-scale combustion testing to municipal waste  
19 incinerators in the United States.

20  
21 **2.0 Summary of study results**

- 22 • A listing of compounds collected at the targeted temperature

23  
24  
25 **3.0 Discussion**

- 26 • Description of the typical municipal incineration process being modeled  
27 including the rationale for selecting targeted temperatures, describe typical  
28 operational parameters, and potential occupational exposures.

29  
30 **4.0 Extrapolation of laboratory test results to the typical municipal incinerator described in**  
31 **section 3.0 (above) for each composite.**

- 32 • A description of the extrapolation
- 33 • A description of any assumptions used
- 34 • Any unique qualitative or quantitative descriptors of the test, the testing  
35 equipment, and the results deemed necessary for informative review of the test  
36 and test results.

37  
38  
39 **5.0 Sensitivity Analysis**

- 40 • Assessment of the impact of variability/uncertainty (quantitative and  
41 qualitative) in each parameter on the modeling results.

42  
43 **6.0 Conclusions**



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**7.0**

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**APPENDIX F**

**QUALITY ASSURANCE PROJECT PLAN (QAPJ) OUTLINE**

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**APPENDIX F**

**COPY OF EPA ORDER INCORPORATING THIS  
ENFORCEABLE CONSENT AGREEMENT**

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**APPENDIX F**

**UNITED STATES  
ENVIRONMENTAL PROTECTION AGENCY**

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

**Docket No. OPPT - ?? xxxxxxxx ??**

18 Under the authority of section 4 of the Toxic Substances Control Act (TSCA), 15 U.S.C.  
19 2603, the United States Environmental Protection Agency (EPA) issues this testing consent order  
20 (Order) to take effect on the date of publication of the notice in the Federal Register announcing the  
21 issuance of this Order. This Order incorporates the enforceable consent agreement (ECA) for the  
22 laboratory-scale incineration testing of the fluoropolymers listed in Appendix A.1 of the ECA.

23  
24  
25 \_\_\_\_\_  
26 Date

25 \_\_\_\_\_  
26 Stephen L. Johnson,  
27 Assistant Administrator  
28 for Prevention, Pesticides,  
29 and Toxic Substances  
30  
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35

1 **Fluoropolymers Incineration Testing Program**

2 DRAFT 10-21-03

3  
4  
5 **I. CHEMICAL SUBJECT TO THE ECA**

6 The test substances being studied in this testing program  
7 are identified in Appendix A. The basis for test substance  
8 selection and the preparation of the composites to undergo  
9 testing are also described in Appendix A.

10  
11 **II. PURPOSE OF TESTING PROGRAM**

12 The purpose of this testing program is to investigate  
13 incineration of designated test substances under  
14 laboratory-scale conditions representative of typical  
15 municipal waste combustor operations in the U.S. to  
16 quantitatively determine potential emission levels of PFOA.

17  
18 **III. SCOPE OF TESTING PROGRAM**

19 The testing program will consist of

- 20  
21 1) conducting the testing listed in Table 1 in accordance  
22 with the test standards specified in Table 1 for the  
23 test substances identified in Appendix A and  
24  
25 2) submitting the reports specified in Table 1 in  
26 accordance with the deadlines set forth in Table 1.

27  
28 **IV. DESCRIPTION OF TESTING PROGRAM**

29 The testing program has 2 phases as follows: Phase I Method  
30 Demonstration and Phase II Incineration Testing.

31  
32 **A. Phase I Method Demonstration**

33 This testing is described in Appendix B. Phase I testing  
34 provides the necessary foundation for Phase II testing.

35  
36 Following completion of Phase I and prior to initiation of  
37 Phase II, the Test Sponsor will provide a brief status  
38 report to EPA demonstrating the progress of testing. This  
39 letter report will provide the result for the overall  
40 recovery across the laboratory-scale thermal reactor system  
41 determined from transport efficiency testing.

42  
43 If this overall recovery for either PFOA or Fluorine is  
44 greater than or equal to 70%, the Test Sponsor will proceed  
45 to Phase II testing.

46  
47 If this overall recovery for neither PFOA nor Fluorine is

1 greater than or equal to 70%, then a Technical Consultation  
2 between the Agency and the Test Sponsor will be held to  
3 reach agreement on whether Phase II testing should proceed  
4 and, if so, on what (if any) revisions to the plans, test  
5 standards, and schedule for Phase II testing to adopt.

6  
7 If this consultation does not result in an agreement to  
8 proceed with Phase II testing, a more complete test report  
9 for Phase I will be submitted to EPA within 60 days of  
10 completion of the Technical Consultation.

11  
12 If Phase II testing is conducted, a more complete test  
13 report for Phase I will be included in the later Phase II  
14 Test Report rather than in a separate report.

15  
16 B. Phase II Incineration Testing

17 As described in Section IV.A, Phase II testing may be  
18 conducted depending on the result of Phase I testing or the  
19 Technical Consultation.

20  
21 Phase II testing is described in Appendix C.  
22  
23

1 **Table 1. Required Testing, Test Standards, and Reporting**

Phase I Testing	Test Standard	Deadline for Final Report <sup>1,3</sup>
Transport Efficiency	See Appendix B	TBD

2

Phase II Testing for Test Substance Composites	Test Standard	Deadline for Final Report <sup>2,4,6</sup>
Elemental Analysis <sup>5</sup>	ASTM D3176 or equivalent as described in Appendix C.1	TBD
Combustion Stoichiometry <sup>5</sup>	See Appendix C.2	TBD
Thermogravimetric Analysis <sup>5</sup>	ASTM E1868 as annotated in Appendix C.3	TBD
Laboratory-scale Combustion Testing <sup>5</sup>	See Appendix C.4 - C.6	TBD
Release Assessment	See Appendix C.6	TBD

3

4 1 Number of months after the effective date of the Order that  
5 incorporates this ECA when final report for this testing is due.  
6 Following completion of Phase I and prior to any initiation of Phase  
7 II, the Test Sponsor will provide a letter report to EPA with the  
8 result for the overall recovery across the laboratory-scale thermal  
9 reactor system determined from Phase I testing.

10 2 If the overall efficiency result from Phase I testing is less  
11 than 70% for both PFOA and Fluorine, then a Technical Consultation will  
12 be held to reach agreement on whether Phase II testing should proceed  
13 and, if so, on what (if any) revisions to adopt to the plans, test  
14 standards, and schedule for Phase II. If such agreement is not  
15 reached, Phase II testing is not required under the Order that  
16 incorporates this ECA.

17 3 A more complete test report for Phase I testing will be submitted  
18 within 60 days of the completion of the Technical Consultation if this  
19 consultation does not result in an agreement to proceed with Phase II  
20 testing. A more complete test report for this testing will be included  
21 in the Phase II test report if Phase II testing is conducted.

22 4 Number of months after initiation of Phase II testing period when  
23 final report for this testing is due. Phase II testing period will be  
24 initiated following Technical Consultation agreement to conduct Phase  
25 II testing or after completion of Phase I testing, whichever is later.

26 5 The results of this testing will be provided in the final report  
27 for Phase II.

28 6 A release assessment will be included in the final report for  
29 Phase II if quantifiable levels of PFOA are found in the exhaust gas  
30 from Phase II combustion testing.

1 **APPENDICES**

2

3 A. Test Substances

4

5 B. Method Demonstration

6

7 C. Incineration Testing



**APPENDIX A. TEST SUBSTANCES**

1. Identification

The four composite test substances for this test program will be prepared from the fluoropolymers 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. 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.

**Table A-1. Test Substance Composites by Type and CAS Number**

Test Substance	Fluoropolymer Type	CAS Number	Associated Monomers
Composite 1 - PTFE (dry non-melt)	PTFE	9002-84-0	TFE
Composite 2 - Dry melt resins	FEP	25067-11-2	TFE, HFP
	PFA	26655-00-5	TFE, PPVE
		31784-04-0	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 Copolymers (dry non-melt)	Fluoroelastomer	9011-17-0	VDF, HFP
	Fluoroelastomer	25190-89-0	TFE, HFP, VDF
	Terpolymers		
	Base resistant elastomers	54675-89-7,	TFE, VDF, P
		27029-05-6	
	Perfluoroelastomers	26425-79-6	TFE, PMVE
CTFE elastomers	9010-75-7	CTFE, VDF	
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	TFE, PPVE
		31784-04-0	TFE, PEVE
THV	25190-89-0	TFE, HFP, VDF	

1  
2 Confidential business information (CBI) regarding the  
3 chemical identity of Low temperature elastomers has been  
4 submitted previously to EPA under separate cover.

5  
6 2. Selection

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

- 10  
11 • Dry melt resins  
12 • Dry non-melt resins and gums  
13 • Aqueous dispersions  
14

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

- 17  
18 • Dry melt resins  
19     1. FEP, PFA, THV, ETFE, HTE  
20  
21 • Dry non-melt resins and gums  
22     2. PTFE  
23     3. Fluoroelastomers  
24  
25 • Aqueous dispersions  
26     4. PTFE, FEP, PFA, THV  
27

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

32  
33 3. Preparation of Composites

34  
35 3.1 Approach

36  
37 A composite mixture of representative fluoropolymers, as  
38 solids, will be prepared for each of the four test  
39 substance composites identified in Table A-1.

40  
41 The polymer samples will be unfilled first quality product  
42 polymer, substantially free of inorganic constituents.  
43 Each sample will be from a representative grade for each  
44 applicable fluoropolymer type from each applicable  
45 producer.  
46

1 For Composite 1 - PTFE, a representative sample of PTFE  
2 from each PTFE producer will be mixed together in equal  
3 proportions across applicable producers to form the  
4 Composite 1 - PTFE.

5  
6 A hypothetical example for Composite Z in Table A-2 below  
7 shows how the other composites will be assembled. In this  
8 example with 4 types across 4 producers, there are 11 x's.  
9 Hence, composite Z would be made up of 11 equal proportions  
10 of the materials indicated with an x.

11  
12 **Table A-2. Compositing Across Producers & Types**

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

13  
14 **3.2 Preparation**

15  
16 Representative samples of each component for each composite  
17 will be sent to the laboratory(ies) in packaging  
18 customarily used for product sample packaging or in  
19 polyethylene, polypropylene, or glass containers.

20  
21 Each composite will be prepared under laboratory conditions  
22 designed to prevent cross-contamination and designed to  
23 assure solids temperatures less than or equal to 60 °C.

24  
25 Following preparation of each composite, the composite will  
26 be placed in a polyethylene, polypropylene, or glass  
27 container.

28  
29 **3.2.1 Composite 1**

30  
31 PTFE is available in powder form. Equal weights of PTFE  
32 powder samples across applicable producers will be mixed  
33 together in dry form to yield Composite 1.

34  
35 **3.2.2 Composite 2**

36  
37 FEP, PFA, THV, ETFE, and HTE dry melt resins are available  
38 in pellet form. Each component of Composite 2 will be  
39 size-reduced (e.g., ground) to produce powder. Equal  
40 weights of the powder form of each component (following the

1 approach in the example for Composite Z in Section 3.1)  
2 will be mixed together in dry form to yield Composite 2.

3  
4 A sample of polyethylene pellets will be size-reduced using  
5 the same technique and equipment to provide a blank. The  
6 resulting polyethylene powder will be archived.

7  
8 3.2.3 Composite 3

9  
10 Fluoroelastomers are available in slab, lump, or sheet  
11 form. Composite 3 will be prepared following one of the  
12 following approaches:

13  
14 a) Equal weights of each component (following the approach  
15 in example for Composite Z in Section 3.1) will be mixed  
16 in a rubber mill to produce a homogenous slab of preset  
17 thickness to yield Composite 3.

18  
19 Or

20  
21 b) Each component of Composite 3 will be cryogenically  
22 cooled (to make the elastomers brittle) and size-reduced  
23 (e.g., ground) to produce powder. Equal weights of the  
24 powder form of each component (following the approach in  
25 the example for Composite Z in Section 3.1) will be mixed  
26 together in dry form to yield Composite 3.

27  
28 A sample of non-fluorinated synthetic rubber will be size-  
29 reduced using the same technique and equipment to provide a  
30 blank. The resulting non-fluorinated rubber sample will be  
31 archived.

32  
33 3.2.4 Composite 4

34  
35 Aqueous dispersions of PTFE, FEP, PFA, and THV are  
36 available as dispersions containing 20 to 60% fluoropolymer  
37 solids by weight. Composite 4 will be prepared following  
38 one of the following approaches:

39  
40 a) Equal weights (on a dry solids basis) of each component  
41 in aqueous dispersion form (following the approach in  
42 example for Composite Z in Section 3.1) will be mixed  
43 together in liquid form. Solids will be separated from  
44 the resulting liquid composite to yield low water content  
45 (i.e., drip free) fine solids.

46  
47 Or

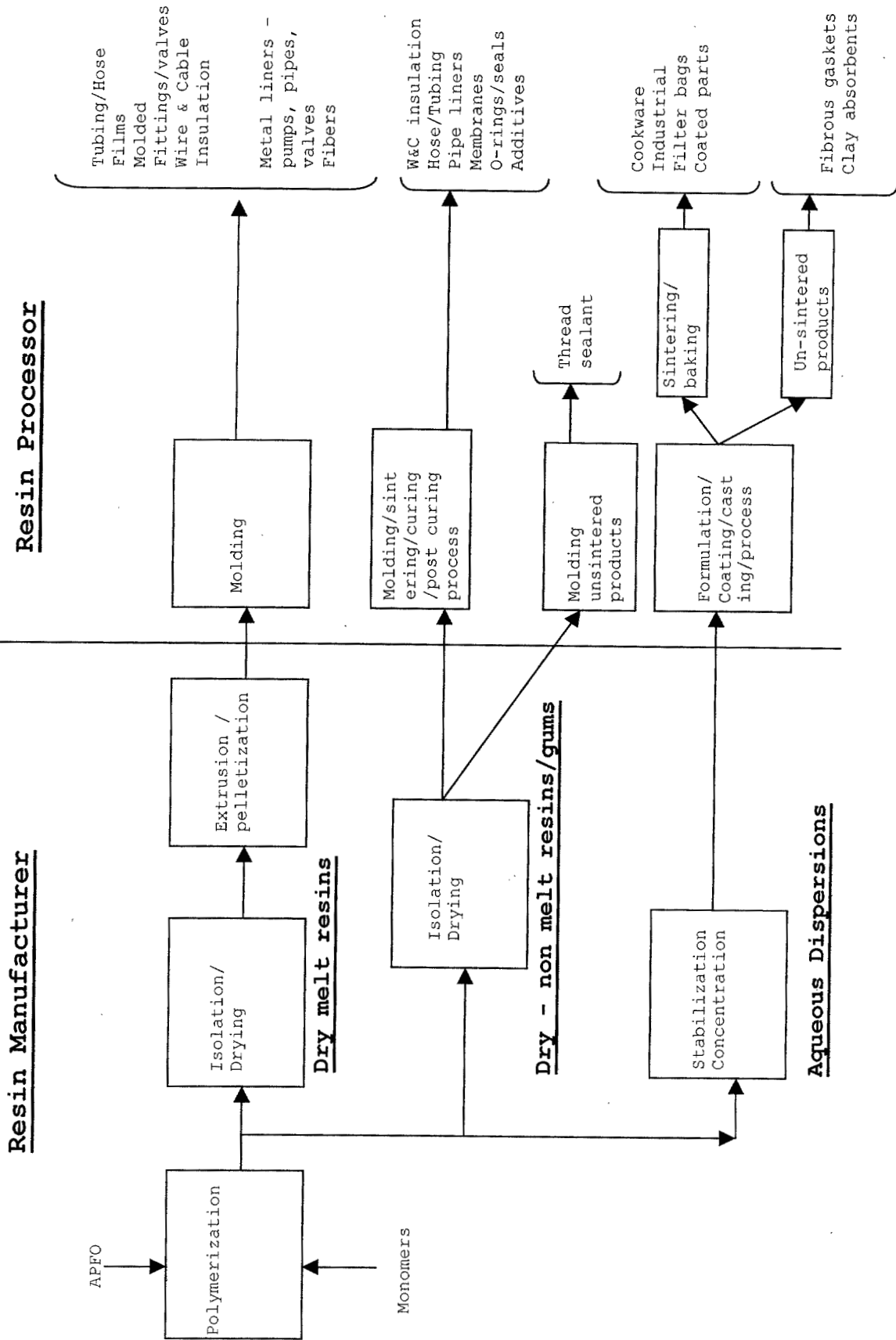
1  
2 b) Solids will be separated from liquid for each component  
3 of Composite 4 to yield low water content (i.e., drip  
4 free) fine solids for each component. Equal weights of  
5 the solids form of each component (following the approach  
6 in the example for Composite Z in Section 3.1) will be  
7 mixed together to yield Composite 4.

8  
9 3.3 Verification

10  
11 In order to assure that composite samples in this testing  
12 program have been made up of clearly identified materials,  
13 the preparation of the composites will include formal Chain  
14 of Custody procedures. A chain of custody form will be  
15 included with each component material going into the  
16 composite to show the identity of the component material  
17 and each transfer of custody from its point of origination  
18 to preparation of the composite. For documentation, the  
19 laboratory preparing a given composite will generate a  
20 report to be submitted to EPA as CBI.

21  
22 Once prepared, each composite will be accompanied by a new  
23 chain of custody until it reaches the incineration testing  
24 facility.

Figure A-1. Fluoropolymer Industry Overview



1 **APPENDIX B. METHOD DEMONSTRATION**

2  
3 Transport Efficiency

4 Quantitative transport of PFOA across the laboratory-scale  
5 thermal reactor system described in Appendix C will be  
6 verified. PFOA of known purity greater than or equal to  
7 97% will be used for this testing. For these transport  
8 tests, 0.5 to 5 mg of PFOA will be gasified at 150 to 250 °C  
9 (based on thermogravimetric analysis of PFOA) with transfer  
10 line and reactor temperatures 0 to 100 °C higher than the  
11 gasification temperature. Sampling and analysis will be  
12 performed to determine quantitation of PFOA and fluorine in  
13 corresponding laboratory-scale thermal reactor system  
14 exhaust gas samples.

15  
16 The amount of PFOA and fluorine determined in this exhaust  
17 gas sample will be compared to the amount of PFOA (as PFOA  
18 and as fluorine) fed to the thermal reactor system. The  
19 amount of PFOA fed to the system will be verified by  
20 weighing the pyroprobe insert cartridge before and after  
21 each experiment.

22  
23 If the resulting transport efficiency for either PFOA or  
24 fluorine is greater than or equal to 70%, then the overall  
25 recovery across the thermal reactor system is also greater  
26 than or equal to 70%.

27  
28 If the resulting transport efficiency for both PFOA and  
29 fluorine is less than 70%, then flexible tubing between the  
30 thermal reactor system and the sample collection apparatus  
31 and/or the high temperature reactor will be examined for  
32 PFOA and/or fluorine. If the flexible tubing is examined,  
33 it will either be extracted using methanol with the  
34 methanol extract being analyzed for PFOA and fluorine, or a  
35 portion of the tubing will be directly subjected to  
36 fluorine analysis. If the reactor is examined, the reactor  
37 will be removed from the system and extracted with  
38 methanol, and the solvent sample will be analyzed for PFOA  
39 and fluorine.

40  
41 In this case, the amount of the analyte (PFOA or fluorine  
42 as applicable) from examination of the tubing and/or the  
43 reactor will be added to the amount of analyte determined  
44 in the exhaust gas sample for comparison with the amount of  
45 analyte fed to the thermal reactor system to determine the  
46 overall recovery across the experimental system.

47

- 1 The thermal reactor system apparatus in this testing
- 2 program is configured such that additional extractions of
- 3 the transfer lines between the pyroprobe and the reactor
- 4 and between the reactor and the vent line (to which the
- 5 flexible tubing is connected) are not feasible.



1 **APPENDIX C. INCINERATION TESTING**

2  
3 Following preliminary testing, combustion testing with  
4 associated exhaust gas sampling and analysis can be  
5 performed. The preliminary testing provides necessary  
6 background information for combustion testing.

7  
8 The standards for preliminary testing are described in  
9 Sections 1, 2, and 3 below. The standard for combustion  
10 testing is presented in Section 4. Section 5 describes the  
11 standard for sampling and analysis for the combustion  
12 testing. Section 6 outlines how results of this testing  
13 program will be reported.

14  
15 1. Elemental Analysis

16 Each test substance composite will undergo elemental  
17 analysis to provide data for stoichiometric calculations.

18  
19 Ultimate analysis (ASTM D3176) is a standard practice for  
20 analysis of coal and coke. It can be used to determine  
21 carbon and hydrogen in a material via analysis of the  
22 gaseous products of its complete combustion, to determine  
23 sulfur, nitrogen, and ash in the material, and to calculate  
24 oxygen in the material by difference.

25  
26 Table C-1 presents the reference standards for test methods  
27 for determination of carbon, hydrogen, nitrogen, sulfur,  
28 chlorine, ash, moisture, and oxygen for the test substance  
29 composites in this program and includes ASTM test methods  
30 referred to in ASTM D3176.

31 **Table C-1. Reference Standards**

Parameter	Test Method
Carbon	ASTM D3178, ASTM D5373, or equivalent
Hydrogen	ASTM D3178, ASTM D5373, or equivalent
Nitrogen	ASTM D3179, ASTM D5373, or equivalent
Sulfur	ASTM D3177, ASTM D4239, or equivalent
Chlorine	Bomb combustion (EPA 5050 or ASTM D808)/chloride ion analysis (ion chromatography or ion selective electrode), ASTM D2361, ASTM D46610B, or equivalent
Ash	ASTM D3174, ASTM D5142, ASTM D482, or equivalent
Moisture	ASTM D3173, ASTM D5142, or equivalent
Oxygen	By difference per ASTM D3176

32  
33  
34 The reference standards will be adapted, as needed, to

1 elemental analysis of the test substance composites in this  
2 testing program. Preparation steps for coal and coke noted  
3 in the ASTM standard test methods in Table C-1 are not  
4 required for application of these methods to the test  
5 substance composites. The sample sizes in the test methods  
6 in Table C-1 will be adjusted as necessary in this testing  
7 program based on available amount of sample.

8  
9 In accordance with ASTM D3176, oxygen will be determined by  
10 difference (i.e, the difference between 100% and the sum of  
11 the other measurements). If fluorine concentration is  
12 analyzed at a different laboratory from that used for the  
13 other elemental analyses, the calculated oxygen level in  
14 the material will be adjusted downward to account for  
15 fluorine.

16  
17 Fluorine (total fluorine) content will be determined via  
18 Wickbold Torch; see Attachment C-1. Based on manufacturing  
19 process knowledge, the levels of total fluorine in the  
20 components of test substance composites are orders of  
21 magnitude higher than the potential trace level of  
22 inorganic fluoride in these materials. Therefore, for this  
23 test program, the total organic fluorine value for each  
24 test substance composite will be assumed to be the same as  
25 the total fluorine value.

26  
27 Manufacturing process knowledge of the polymers will be  
28 used to review the elemental analysis results and to form  
29 the basis for interpreting non-detects. For example, if  
30 the elemental analysis result for a non-chlorinated or ash-  
31 free polymer is non-detect with a quantitation limit of  
32 0.1%, then the analytical result will be replaced with 0.

33  
34 Additionally, ASTM D3176 may be used as a reference to  
35 convert elemental analysis results to a basis (e.g., dry)  
36 other than on which the results are reported by the  
37 elemental analysis laboratory.

38  
39 2. Combustion Stoichiometry

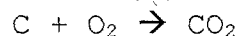
40 The results of the elemental analysis will form the basis  
41 for combustion stoichiometry calculations needed to help  
42 establish combustion test conditions.

43  
44 First, the weight percent values from elemental analysis  
45 (see Section 1) are converted to molar quantities.

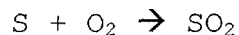
46  
47 Second, based on Chapter 3 of Combustion Fundamentals for

1 Waste Incineration (American Society of Mechanical  
2 Engineers, 1974), the reaction products for these molar  
3 quantities are calculated assuming complete combustion with  
4 the following behavior:

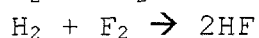
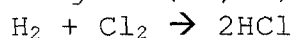
5  
6 a) All carbon (C) in feed converts to carbon dioxide (CO<sub>2</sub>)



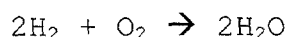
8  
9 b) All sulfur (S) in feed converts to sulfur dioxide (SO<sub>2</sub>)



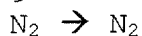
11  
12 c) The halogens (Cl, F) in feed convert to hydrogen halides



15  
16 d) Hydrogen (H) present in feed in excess of that  
17 required to yield products in item c) above will be  
18 converted to water



20  
21 e) Nitrogen (N) from feed or air is emitted as molecular  
22 nitrogen



24  
25 Third, with these rules, the balanced chemical reaction for  
26 combustion of a compound can be written.

27  
28 For a hydrocarbon like methane (CH<sub>4</sub>), the resulting reaction  
29 equation is



32  
33 Note that the term feed in the preceding rules (a through  
34 e) includes both material being combusted and the fuel  
35 source of hydrogen such as methane or methanol.  
36 Additionally, stoichiometric calculations as described  
37 above presume that the compounds undergoing combustion are  
38 essentially free of inorganic constituents.

39  
40 The amount of oxygen needed for the overall combustion  
41 reaction for a feed is called the stoichiometric oxygen  
42 level. The actual oxygen level for combustion is generally  
43 set in terms of excess oxygen or excess air.

44  
45 Additional stoichiometric calculations will be performed as  
46 needed to set and adjust experimental conditions for  
47 combustion testing; see Section 4.3.

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46  
47

### 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 at 25°C/minute using 1 to 5 mg samples. ASTM E1868 as annotated will be used as the guideline for this analysis; see Attachment C-3.

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.

The temperature for complete gasification for each test substance composite will be considered in establishing conditions for the gasification section (pyroprobe section) of the experimental apparatus for the combustion tests; see Section 4.3.

### 4. Combustion Testing

#### 4.1 Test Objective

The objective of this combustion testing program is to investigate incineration of the test substances under laboratory-scale conditions representative of typical municipal waste combustor operations in the U.S. to quantitatively determine potential emission levels of PFOA.

#### 4.2 Experimental Apparatus

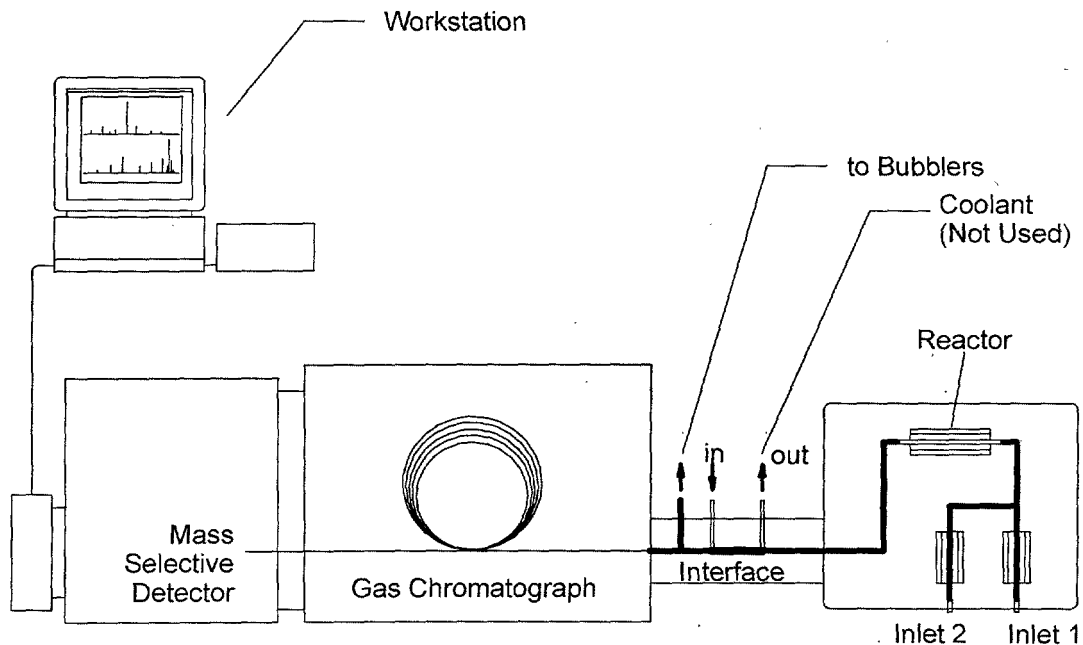
Combustion testing will make use of the Advanced Thermal Reactor System (ATRS) at the University of Dayton Research Institute (UDRI). The ATRS is a laboratory-scale, non-flame, batch-charged, continuous flow thermal reactor system. The use of a non-flame thermal reactor system gives a conservative representation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

In the ATRS, the test sample is gasified and transported to a high temperature fused silica reactor. In the high

1 temperature reactor, the sample vapors are subjected to  
2 controlled conditions of residence time, temperature, and  
3 excess air. Combustion products (section 5) will be  
4 collected for quantitative analysis.

5  
6 A schematic of the ATRS as configured for this test program  
7 is shown in Figure C-1.

8  
9 **Figure C-1. Schematic of ATRS for this Test Program**



10  
11

12 The ATRS consists of a reactor assembly and in-line gas  
13 chromatograph/detector system connected via an interface.  
14 The reactor assembly consists of a thermally insulated  
15 enclosure housing the sample introduction, reactor, and  
16 transfer line systems.

17

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

23

24 During combustion tests, the transfer line between the  
25 pyroprobe and the reactor is heated and maintained above  
26 200 °C. The reactor is housed within its own small tube  
27 furnace and may be independently heated to as high as 1200  
28 °C. (Actual conditions for this test program are presented

1 in Section 4.3.) The transfer line from the reactor to the  
2 interface is heat traced to greater than 200 °C to prevent  
3 cool regions where reactor products could otherwise be lost  
4 through condensation.

5  
6 The interface routes the combustion exhaust gas to the in-  
7 line gas chromatograph (GC) and mass selective detector  
8 (MSD) or to sample collection for off-line analysis. For  
9 combustion testing in this test program, the interface will  
10 also be maintained above 200 °C.

11  
12 Exhaust gas sampling and analysis (including use of the in-  
13 line GC and MSD) are described in Section 5.

#### 14 15 4.3 Combustion Test Experimental Conditions

16  
17 The test substance composites will be subjected to  
18 laboratory-scale incineration using the experimental  
19 apparatus described in Section 4.2.

##### 20 21 4.3.1 Combustion Air

22  
23 Synthetic air (mixture of 21% oxygen and 79 % nitrogen)  
24 will be used in place of compressed air to prevent  
25 potential interference in the experimental system due to  
26 background levels of CO<sub>2</sub> in compressed air.

##### 27 28 4.3.2 Fuel

29  
30 Methanol will be used as needed as a supplemental fuel to  
31 ensure the presence of sufficient hydrogen to convert  
32 fluorine to hydrogen fluoride (HF) and chlorine to hydrogen  
33 chloride (HCl).

34  
35 As noted in *Municipal Solid Waste in the United States:  
36 2000 Facts and Figures* (EPA, 2002), paper and paper  
37 products (made from wood) make up the largest component of  
38 municipal solid waste (MSW). The sum of paper and paper  
39 products with wood in MSW makes up over 30% of MSW.

40  
41 During the 19<sup>th</sup> century, methanol was produced from wood and  
42 was known as wood alcohol. Therefore, methanol can be used  
43 in this experimental program as a surrogate for the paper  
44 and wood fraction of MSW.

45  
46  
47

1 4.3.3 Operating Conditions

2  
3 The target operating conditions for the high temperature  
4 reactor during the combustion tests are presented in Table  
5 C-2.

6  
7 **Table C-2. Combustion Test Target Operating Conditions**

Test Material	PTFE composite	Dry melt resin composite	Fluoroelastomer composite	Aqueous dispersion composite
Temperature	900 °C	900 °C	900 °C	900 °C
Residence Time	2 sec	2 sec	2 sec	2 sec
O <sub>2</sub> conc. in Exhaust Gas	10%	10%	10%	10%
H <sub>2</sub> O conc. in Exhaust Gas	14%	14%	14%	14%
No. of runs	3	3	3	3

8  
9 These conditions are representative of typical operating  
10 conditions for the high temperature zone of municipal waste  
11 combustors (MWCs) in the U.S. The temperature was selected  
12 to conservatively represent the low end of normal MWC  
13 operating temperatures and is also less than typical  
14 secondary chamber operating temperatures for medical waste  
15 incinerators in the U.S. See Attachment C-4 for supporting  
16 information.

17  
18 The expected sample size is in the range of 1 to 3 mg. The  
19 actual sample size, the gasification rate (determined from  
20 TGA), and the excess air will be adjusted to assure that  
21 the oxygen level in the exhaust will be greater than or  
22 equal to the concentration in Table C-2 to be  
23 representative of typical MWC conditions.

24  
25 Temperature and residence time values in Table C-2 will be  
26 fixed setpoints for the experiment. Fuel supply and excess  
27 air will be adjusted as needed to approach the H<sub>2</sub>O  
28 concentration target in Table C-2. Excess air will be  
29 further adjusted as needed based on fuel supply. Note that  
30 O<sub>2</sub> and H<sub>2</sub>O exhaust gas concentration values will be  
31 determined by calculation in planning the experiment.

32  
33 The temperature in the pyroprobe section will be maintained  
34 at 750 °C or as needed to assure this section is 50 to 100

1 °C above the highest temperature for complete gasification  
2 across the test materials as determined from the TGA  
3 experiments earlier in the test program; see Section 3.  
4 This is necessary to assure complete gasification of the  
5 sample of test material and a common set of experimental  
6 conditions across the test materials during combustion  
7 testing.

8  
9 4.4 Blanks

10  
11 *[under development]*

12  
13 4.5 Process Monitoring

14  
15 Monitored process parameters and associated recording  
16 frequencies for each combustion test are presented in Table  
17 C-3.

18  
19 **Table C-3. Combustion Test Monitoring**

Parameter	Recording Frequency
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

20  
21 Temperature-Inlet 1 will be recorded on a log sheet at the  
22 end of the temperature ramp-up for gasification to monitor  
23 the pyroprobe final temperature.

24  
25 Recording the other values on a log sheet with the  
26 frequency noted in Table C-3 assures that each parameter is  
27 recorded at least once every 15 minutes because the  
28 expected duration of each combustion test is approximately  
29 5 to 15 minutes.

30  
31 The flow rate of the exhaust gas routed to the bubblers  
32 (described in Section 5.2) will be determined based on the  
33 flow measurements listed in Table C-3.

34  
35 The amount of material fed to the system will be verified  
36 by weighing the pyroprobe insert cartridge before and after



1 each experiment.

2

3 Exhaust gas oxygen concentration will be calculated, based  
4 on measured total input flow rate of synthetic air and fuel  
5 assuming complete combustion.

6

7 5. Sampling and Analysis

8

9 5.1 Exhaust Gas Monitoring

10

11 Combustion exhaust gas will be monitored for carbon  
12 monoxide (CO) and CO<sub>2</sub> via the in-line GC using a molecular  
13 sieve column and MSD or via off-line analysis of exhaust  
14 gas collected in Tedlar® bags.

15

16 5.2 Exhaust Gas Sampling

17

18 Gas samples for off-line analysis will be collected from a  
19 vent line off the interface. Flexible tubing will connect  
20 the vent line and a set of bubblers.

21

22 Gas absorption via these bubblers will provide aqueous  
23 solution to analyze for the parameters in Section 5.3. Two  
24 to four bubblers (low pressure drop impingers) in series  
25 will be used. Each bubbler will contain a predetermined  
26 amount of aqueous solution. The temperature of the gas  
27 exiting the last bubbler will be monitored.

28

29 An additional empty bubbler will be added to the front end  
30 of this series of bubblers to serve as a knock-out pot if  
31 stoichiometric calculations (see Sections 2 and 4.3) for  
32 the test substance or experience from earlier combustion  
33 testing with test substance indicate that greater than 10  
34 mL of water will be produced during the combustion test of  
35 a given test substance.

36

37 Upon completion of sample collection, the amounts in each  
38 bubbler will be determined by observation or by weight, and  
39 the contents of the bubblers will be composited for  
40 subsequent analysis. Additionally, the flexible tubing  
41 will be rinsed with 5 mL of HPLC water to collect potential  
42 condensate in the tubing. This rinsate may be combined  
43 with the bubbler composite prior to analysis.

44

45 A minimum of 60 mL of bubbler aqueous solution is expected  
46 from each combustion test. Of this, a minimum of 45 mL  
47 will be sent for PFOA analysis, a minimum of 5 mL will be

1 sent for fluoride (and chloride) ion analysis, and a  
2 minimum of 2 mL will be sent for total fluorine analysis.

3  
4 Bubbler aqueous solutions will be conveyed to analytical  
5 laboratories in a polyethylene, polypropylene, or glass  
6 containers.

### 7 8 5.3 Exhaust Gas Analysis

#### 9 10 5.3.1 Fluoride & Chloride Ion

11  
12 A portion of the composite aqueous solution sample from  
13 each combustion test collected as described in Section 5.2  
14 will be analyzed for fluoride ion and for chloride ion via  
15 ion chromatography.

#### 16 17 5.3.2 Total Fluorine

18  
19 A portion of the composite aqueous solution sample from  
20 each combustion test collected as described in Section 5.2  
21 will be analyzed for total fluorine via Wickbold Torch at a  
22 qualified laboratory; see Attachment C-1.

#### 23 24 5.3.3 PFOA

25  
26 A portion of the composite aqueous solution sample from  
27 each combustion test collected as described in Section 5.2  
28 will be analyzed for PFOA via LC/MS/MS at a qualified  
29 commercial laboratory in accordance with "Method of  
30 Analysis for the Determination of Ammonium  
31 Perfluorooctanoate (APFO) in Water Revision 1" (Docket ID  
32 OPPT-2003-0012-0040) as revised for exhaust gas bubbler  
33 sample analysis for PFOA; see Attachment C-5.

34  
35 Due to background levels of PFOA, the analytical laboratory  
36 will only report numerical values for PFOA concentration in  
37 the aqueous solution greater than or equal to the limit of  
38 quantitation (LOQ). This is required to assure that the  
39 reported concentration value is attributable to the sample  
40 rather than to background.

### 41 42 5.4 Test Substance Sampling & Analysis

43  
44 See Section 1.

## 45 46 6. Reporting of Results

1 6.1 Elemental Analysis Results

2  
3 The results of elemental analysis of the test substance  
4 composites (as noted in Section 1) will be reported.

5  
6 6.2 Combustion Stoichiometry Results

7  
8 Example combustion stoichiometry (as noted in Section 2)  
9 calculations will be included in the test report.

10

11 6.3 TGA Results

12

13 TGA results for test substance composites (as noted in  
14 Section 3) will be included in an appendix to the test  
15 report.

16

17 6.4 Combustion Test Results

18

19 6.4.1 Process Monitoring

20

21 Process monitoring data (as noted in Section 4.5) recorded  
22 for each combustion test will be reported in tabular form.

23

24 6.4.2 Exhaust Gas Monitoring

25

26 Exhaust gas monitoring results will be reported as the  
27 average (integrated value) for each combustion test. CO  
28 will be reported in terms of parts per million by volume  
29 (ppmv). CO<sub>2</sub> will be reported in terms of percent by volume  
30 (%).

31

32 6.4.3 Exhaust Gas Results

33

34 Results of analyses noted in Section 5.3 will be reported  
35 for each combustion test.

36

37 The analytical result for each analyte in Section 5.3 will  
38 be reported in terms of concentration (mass per volume) in  
39 the aqueous solution. For each analyte, this value will be  
40 used with the associated exhaust gas volume to compute an  
41 exhaust gas concentration.

42

43 6.4.3.1 Fluoride and Chloride

44

45 Fluoride ion and chloride ion in the exhaust gas will be  
46 reported in terms of concentration (mass of ion per volume  
47 of exhaust gas) in the gas and on the basis of mass of ion

1 per mass of test substance. The corresponding hydrogen  
2 halide value for each will also be computed and reported  
3 for reference.

4  
5 The fluorine ion value is also the total inorganic fluorine  
6 value.

7  
8 6.4.3.2 Total Fluorine and Total Organic Fluorine

9  
10 Total fluorine in the exhaust gas will each be reported in  
11 terms of concentration (mass of fluorine per volume of  
12 exhaust gas) in the gas and on the basis of mass of  
13 fluorine per mass of test substance.

14  
15 Total organic fluorine will be determined by difference  
16 between total fluorine and total inorganic fluorine.

17  
18 6.4.3.3 PFOA

19  
20 PFOA in the exhaust gas will be reported in terms of  
21 concentration in the gas (mass of PFOA per volume of  
22 exhaust gas) and on the basis of mass per mass of test  
23 substance.

24  
25 6.5 Test Substance Results

26  
27 Elemental compositions will be reported as indicated in  
28 Section 6.1 above.

29  
30 6.6 Release Assessment

31  
32 In the event that PFOA is found in the exhaust gas at a  
33 concentration above the LOQ (for the matrix) for the  
34 average of three runs for a given test substance composite,  
35 then the potential for release from full-scale waste  
36 incineration of the subject material will be assessed to  
37 inform the basis for possible next steps.

38  
39 This assessment will consider a number of factors such as

- 40  
41 • test program-determined PFOA emission factor,  
42 • estimated amounts of subject material in feed to full-  
43 scale waste incinerators, and  
44 • degree of post-combustion air pollution control (e.g.,  
45 use and effectiveness of carbon adsorption).

46  
47

WORKING DRAFT FOR DISCUSSION  
FOR DELIBERATIVE PURPOSES ONLY

DO NOT CITE OR QUOTE  
10-21-03

- 1 Attachments
- 2
- 3 Attachment C-1 Wickbold Torch
- 4
- 5 Attachment C-3 TGA:Annotated ASTM E1868
- 6
- 7 Attachment C-4 Waste Incineration and Operating Conditions
- 8
- 9 Attachment C-5 PFOA Analysis