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OPPT-2003-0071-0055



Rich Leukroth

01/15/04 05:19 PM

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cc: John Blouin/DC/USEPA/US@EPA, Greg Fritz/DC/USEPA/US@EPA,
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Subject: January 21 Fluoropolymer ECA Discussions

The next Fluoropolymer Incineration ECA development teleconference is scheduled for Wednesday, January 21st between 7 a.m. and 9 a.m. Eastern Standard Time.

Charles Auer has asked that the Drafting Committee have a document reflecting our progress in developing this ECA available to the Interested Parties for the January Workgroup and Plenary meetings. As a result I have manually compiled what I believe is the most recent versions (i.e., sans any changes discussed on 1/13/04) of the appendices into a single pdf file. In addition, I have incorporated comments received from the 1/13/04 discussions into Draft #6 of the ECA document. These files are attached below.

We will need to focus on accomplishing as much as possible to resolve the outstanding issues in the ECA document in the two hour call-in time. To the extent possible please be prepared to discuss specific changes and be willing to acknowledge concurrence to move forward to the Interested Parties with ECA document text by the end of the meeting. Where a difference in views on outstanding points remain, the Drafting Committee will need to reach agreement on the summary of progress statement that will be included in the document transmitted to the Interested Parties.

Charlie has instructed me to distribute the draft ECA to the Interested Parties as it stands on January 22, 2004.

TENTATIVE AGENDA

- 1) Introductions
- 2) ECA document:
 resolve follow-on issues and
 establish a tentative schedule for testing in Table 1
- 3) ECA appendices:
 resolve outstanding issues and
 resolve existing holes in the appendices (e.g., D.4)
- 4) Prepare for the January 27-29 meetings
- 6) Other discussions (to be determined by the group)
- 7) Next Steps

TELECONFERENCE CALL INFO:

Note: Robert Giraud has agreed to establish the telephone conference lines via AT&T for this meeting and will be providing the call-in details to us in a subsequent e-mail before Wednesday (1/21/04).

ATTACHMENTS



1) ECA Document Draft #6

ECA_FluoroIncinc_dft_1_21_04.p



2) Compilation of ECA Appendices

ECA_Appendices_1_15_04.pr

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DRAFT #6 TO PFOA ECA PROCESS DRAFTING COMMITTEE

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

Docket No. OPPT - [2003] - [00071]

**[DRAFT 1/21/04]
[Month Year]**

NOTE TO DRAFTING COMMITTEE:

This 1/21/04 draft #6 incorporates changes as discussed at the 1/13/04 meeting.

- 1) **Red text** (which shows up as shaded text in the pdf file) indicates places where suggested revisions, new text, and/or carry over text from prior drafts has been inserted. Strikeout shows text to be deleted. In places where there were no reservations voiced at the last discussions, prior red text and strikeouts are now incorporated as document text.
- 2) ***** FOLLOW-ON Discussion** indicates areas for further discussion. This includes original text, proposed changes, and, in some cases, alternative text. Additionally, [.text..] provides a summary / overview of progress made and/or differing views.

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ENFORCEABLE CONSENT AGREEMENT FOR THE LABORATORY SCALE
INCINERATION TESTING OF FLUOROPOLYMERS

Docket No. OPPT-2003-0071

TABLE OF CONTENTS

I.	<u>INTRODUCTION</u>	1
II.	<u>TEST SUBSTANCES</u>	2
III.	<u>OBLIGATION OF SIGNATORY COMPANIES</u>	3
IV.	<u>PRINCIPAL TEST SPONSORIES</u>	4
V.	<u>PURPOSE OF THE TESTING PROGRAM</u>	4
VI.	<u>SCOPE OF THE PROGRAM</u>	5
VII.	<u>DESCRIPTION OF THE TESTING PROGRAM</u>	5
VIII.	<u>PHASE I TECHNICAL CONSULTATION</u>	6
IX.	<u>STANDARDS FOR CONDUCTING TESTING</u>	7
X.	<u>STUDY PLANS AND QUALITY ASSURANCE ACTION PLANS (QAP)P</u>	9
XI.	<u>MODIFICATIONS TO ENFORCEABLE CONSENT AGREEMENT</u>	9
XII.	<u>FAILURE TO COMPLY WITH THE ENFORCEABLE CONSENT AGREEMENT</u>	9
XIII.	<u>EPA MONITORING OF ENFORCEABLE CONSENT AGREEMENT TESTING</u>	11
XIV.	<u>SUBMISSIONS TO EPA AND CONFIDENTIALITY OF INFORMATION</u>	11
XV.	<u>PUBLICATION AND DISCLOSURE OF TEST RESULTS</u>	13
XVI.	<u>OTHER RESPONSIBILITIES OF THE COMPANIES</u>	13
XVII.	<u>SEVERABILITY OF ENFORCEABLE CONSENT AGREEMENT PROVISIONS</u>	14

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

-i-

XVIII. <u>FINAL AGENCY ACTION</u>	14
XIX. <u>PUBLIC RECORD</u>	14
XX. <u>EFFECTIVENESS</u>	14
XXI. <u>RIGHTS OF THE COMPANIES</u>	15
XXII. <u>RESERVATION OF RIGHTS BY COMPANIES</u>	15
XXIII. <u>IDENTITY OF THE COMPANIES</u>	16
XXIV. <u>SIGNATURES</u>	17
Table 1. REQUIRED TESTING, TEST STANDARDS, REPORTING AND OTHER REQUIREMENTS FOR THE LABORATORY-SCALE INCINERATION TESTING OF FLUOROPOLYMERS	22

APPENDICES

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

- B. Testing Standard with Annotations as Appropriate
 - B.1 ASTM E 1868-02 Loss-On-Drying by Thermogravimetry

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

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FLUOROPOLYMER ECA DRAFTING COMMITTEE

ii

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

- E. Outlines for Interim Progress Reporting and Release Assessment Report
 - E.1 Interim Progress Reporting
 - E.2 Release Assessment Report

- F. Copy of the EPA Order

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FLUOROPOLYMER ECA DRAFTING COMMITTEE

-iii-

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

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1 Workgroup during subsequent meetings. On [Month/Date], 2003, the Fluoropolymer Technical
2 Workgroup acknowledged that this testing program had sufficient merit for consideration by the Plenary
3 Group. On [Month/Date], 2003, the Plenary Group discussed the merit of this testing program and
4 recommended that EPA consider entering into an ECA with test sponsors. The official record for the
5 development of this ECA, including the public version, is established under EPA docket control number
6 [OPPT-2003-0012]. The procedures for ECA negotiations are described at 40 CFR 790.22(b). The
7 official record for the testing conducted under this ECA is Docket No. OPPT-2003-0071
8
9

10 ***** FOLLOW-ON DISCUSSION**

11 [On 1/13/04 FMG requested to delete the dry non-melt PTFE resin composite as a test
12 substance in Part II and Appendices. FMG proposed PTFE be incorporated into the
13 remaining three composites. (see also Part XVI of the ECA for further detail.) On 1/21/04
14 EPA indicated that because the fluoropolymer commercial products are manufactured in four
15 forms and since this formed the basis for the composite selection criteria which was embraced
16 by the Workgroups and Plenary it is unreasonable to make this change.]
17

18 **II. TEST SUBSTANCES**

19
20 For the purposes of testing under this ECA the chemicals listed in Appendix A.1¹ will be
21 combined to form four composites (see Appendix A.3 and A.4). These four composites are
22 considered the subject test substances under this ECA. These composites are representative of
23 fluoropolymer products manufactured by the Companies that are currently available in the marketplace.
24 The Companies will provide the fluoropolymers specified in Appendix A.1 for incorporation into the
25 composites that will be tested under this ECA.² Criteria for the selection of each composite to be
26 tested under this ECA are described in Appendix A.2 of this ECA¹. The component composition of
27 each composite is described in Appendix A.3 of this ECA¹. The four composites to be tested are
28 defined for purposes of this ECA as:
29

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

¹ There is a Public and CBI version of Appendices A.1, A.3 ~~A.2, 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).

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

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- 1 (B) Dry Melt Fluoropolymer Resin Composite: (containing: 1-Propene,
2 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No.
3 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3-
4 [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No.
5 26655-00-5; Ethene, tetrafluoro-, polymer with
6 trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; 1-Propene,
7 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and
8 tetrafluoroethene, CAS No. 25190-89-0; 1-Hexene,
9 3,3,4,4,5,5,6,6-nonafluoro-, polymer with ethene and
10 tetrafluoroethene, CAS No. 68258-85-5; and, 1-Propene,
11 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene,
12 CAS No. 35560-16-8),
13
- 14 (C) Dry Non-Melt Fluoroelastomer Gum Composite: (containing: 1-
15 Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene,
16 CAS No. 9011-17-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer
17 with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0;
18 1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene,
19 CAS No. 54675-89-7; 1-Propene, polymer with tetrafluoroethene,
20 CAS No. 27029-05-6; Ethene, tetrafluoro-, polymer with
21 trifluoro(trifluoromethoxy) ethene, CAS No. 26425-79-6; and, Ethene,
22 chlorotrifluoro-, polymer with 1,1-difluoroethene, CAS No. 9010-75-
23 7; and ??generic name??. **Accession No. ??????**, and
24
- 25 (D) Aqueous Fluoropolymer Dispersions Composite: (containing: Ethene,
26 tetrafluoro-, polymer with trifluoro(pentafluoroethoxy) ethene, CAS
27 No. 31784-04-0; Ethene, tetrafluoro-, homopolymer, CAS No.
28 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with
29 tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-
30 heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene,
31 CAS No. 26655-00-5; Ethene, tetrafluoro-, polymer with
32 trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0; and 1-
33 Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and
34 tetrafluoroethene, CAS No. 25190-89-0.
35

36 The procedure for constructing each composite is described in Appendix A.4 to this ECA¹.
37 The polymer components for each composite will be unfilled first quality product polymer, substantially

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1 free of inorganic constituents. Each component of the four composites to be tested under this ECA will
2 be accompanied by a certificate of analysis showing it to meet applicable product specifications.
3

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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 XII of this ECA). In the
11 event that one or more of the Companies are in violation as described above then the remaining
12 Companies will inform EPA of the problem and request an EPA determination on how to proceed with
13 the testing program described under this ECA. Each Company required to contribute to a particular
14 composite is obligated to complete the testing required by this ECA for that composite. A Company
15 shall not be responsible for any failure to perform its obligation under this ECA that is caused by
16 circumstances beyond its control, that the Company could not have prevented through the exercise of
17 due diligence. Under such circumstances the Company will consult with EPA to reach agreement on
18 what modifications, if any, are needed in the test plan or scope of testing (see Part X of this ECA
19 regarding 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 time periods that begin on the
25 effective date, will be treated as beginning on that publication date.

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

29
30 The Companies have identified the Fluoropolymer Manufacturers Group (FMG), to
31 communicate with EPA about schedules, study plans, protocols, test standards, and other aspects of
32 the testing program. EPA and the Companies agree that FMG has no legal responsibility for complying
33 with this ECA. Responsibility for complying with the ECA rests at all times with the Companies.

34
35
36 **V. PURPOSE OF THE TESTING PROGRAM**

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

6
7 EPA believes that these incineration studies of fluoropolymers will develop data needed by the
8 Agency to determine whether municipal and/or medical waste incineration of fluoropolymers is a
9 potential source of PFOA that may contribute as a pathway to environmental and human exposures.
10 The data may also be used to inform screening level human and environmental exposure assessments.
11 In addition, the data may also be used by other Federal agencies (e.g., the Agency for Toxic
12 Substances and Disease Registry (ATSDR), the National Institute for Occupational Safety and Health
13 (NIOSH), the Occupational Safety and Health Administration (OSHA), and the Consumer Product
14 Safety Commission (CPSC), the Food and Drug Administration (FDA)) in assessing chemical risks and
15 in taking appropriate actions within their programs. It is intended that the data generated under this
16 ECA will identify whether the incineration of fluoropolymers contributes to the sources and pathways of
17 environmental and human exposure to PFOA.

18
19
20 **VI. SCOPE OF THE PROGRAM**

21
22 The scope of this testing program is described in Parts VII and VIII below and will consist of
23 the testing listed in Table 1 in accordance with the test standards specified in Table 1 and described in
24 Appendix B.1 and C.1 - C.2 as annotated by Appendix D.1- D.4 to this ECA ("Test Standards") and
25 submitting the reports and documents specified in Table 1 in accordance with the deadlines set forth in
26 Table 1 and described in Appendices C.1 - C.2 and E.1- E.2.

27
28
29 **VII. DESCRIPTION OF THE TESTING PROGRAM**

30
31 The program has two segments as follows: Phase I PFOA Transport Testing and Phase II
32 Fluoropolymer Incineration Testing.

33
34 A. Phase I PFOA Transport Testing: Phase I will consist of quantitative transport
35 efficiency testing for PFOA. Phase I testing for PFOA transport efficiency is specified in the Phase I
36 PFOA Transport Testing segment of Table 1 and described in Appendix C.1 as annotated by
37 Appendix D.1 and D.2. At the conclusion of Phase I testing, the Companies, will provide EPA with a
38 letter report summarizing the results. In the event that the transport efficiency of PFOA or total fluorine

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1 (as determined by the formulas in Appendix C.1) is equal to or greater than 70%, testing will proceed
2 to Phase II Fluoropolymer Incineration Testing. In the event the transport efficiency of PFOA or total
3 fluorine (as determined by the formulas in Appendix C.1) is less than 70%, the Companies will initiate a
4 technical consultation with EPA (see Part VII. B. and Part VIII of this ECA).

5
6 **B. Phase II Fluoropolymer Incineration Testing:** This testing, specified in the Phase II
7 Fluoropolymer Incineration Testing segment of Table 1 and described in Appendix C.2.1 - C.2.6 as
8 annotated by Appendices B.1, D.1, D.2, and E.2; and will include the following for each fluoropolymer
9 composite to be tested under this ECA: 1) elemental analysis, 2) combustion stoichiometry, 3)
10 thermogravimetric analysis, 4) laboratory-scale combustion testing, and, 5) if required under this ECA,³
11 release assessment reporting.

12
13
14 **VIII. PHASE I TECHNICAL CONSULTATION**

15
16 **A.** Following completion of Phase I and prior to the initiation of Phase II, the Companies will
17 submit a letter report to EPA with the results for the recovery across the laboratory-scale thermal
18 reactor system, as determined from Phase I testing.

19
20 **B.** If the recovery for either PFOA or Total Fluorine (as determined by the formulas in
21 Appendix C.1) is greater than or equal to 70%, the Companies will proceed to Phase II testing.

22
23 **C.** If the recovery for both PFOA and Total Fluorine (as determined by the formulas in
24 Appendix C.1) is less than 70%, a Technical Consultation will be held between the Companies and
25 EPA. The objective of the Technical Consultation will be to reach agreement on how to proceed. The
26 technical consultation will review the outcomes of the Phase I PFOA Transport Efficiency Testing,
27 discuss the feasibility of proceeding with Phase II Testing as described in this ECA, and discuss
28 whether additional modifications are needed to the test standards and/or protocols described in
29 Appendices B, C and D for Phase I PFOA Transport Testing and/or Phase II Fluoropolymer
30 Incineration Testing. Specifically, the technical consultation will address: (1) whether the data from the

³ In the event that Phase II Fluoropolymer Incineration Testing identifies measurable levels of PFOA (where measurable PFOA is defined to be at or above the limit of quantitation (LOQ) as defined in Appendix D.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 full-scale municipal and/or medical waste incinerator operations in the United States.

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1 Phase I PFOA Transport Testing segment provide a sufficient basis for conducting the laboratory-scale
2 incineration testing specified in the Phase II Fluoropolymer Incineration Testing segment; (2) the nature
3 and scope of any additional Phase I work that may be required prior to the commencement of Phase II
4 Testing and reporting (e.g., modifications to the Advanced Thermal Reactor System) as described in
5 Part VII. B. of this ECA), and/or (3) the nature and scope of modifications to the protocols and test
6 standards for Phase I and/or Phase II testing, or the identification of additional testing, that may be
7 needed to complete the testing under this ECA.

8
9 Possible outcomes of the Technical Consultation include, the following:

- 10 1. An agreement to conduct additional Phase I testing,
11 and the schedule and standards for such testing, to
12 inform whether and under what conditions to conduct
13 Phase II testing.
- 14
15 2. An agreement to proceed into Phase II testing with or
16 without agreed-to modifications to plans, test standards
17 and schedules for Phase II testing.
- 18
19 3. An agreement to conduct such other testing, and the
20 schedule and standards for such testing, in Phase II that
21 the Companies and EPA agree may be appropriate, in
22 light of Phase I results, to assist in determining the
23 potential for release of PFOA from fluoropolymers
24 during waste incineration.
- 25
26 4. No agreement on a path forward, in which case the Companies' obligations to
27 conduct testing or reporting beyond Phase I PFOA Transport Testing as
28 described in this ECA are terminated.

29
30 D. EPA shall place in the docket (OPPT-2003-0071) a summary of any Technical
31 Consultation that is held under this paragraph. In the event modifications to the testing program are
32 agreed to, EPA and the Companies will revise this ECA, as well as Table 1 and the Appendices, as
33 appropriate.

34
35
36 **IX. STANDARDS FOR CONDUCTING TESTING**

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

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

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

22 ***** FOLLOW-ON DISCUSSION POINT:**

23 **[SUMMARY: FMG maintains that the University of Dayton laboratory can not comply with**
24 **GLPS requirements for testing under this ECA. In addition, FMG expresses concern about**
25 **duplication between QAPjP and study plan requirements. EPA maintains that: 1) study plan(s)**
26 **are required, 2) all studies must be conducted in accordance with GLPS and 3) separate**
27 **QAPjP(s) must be submitted. EPA noted that cut and paste from ECA protocols etc. can be**
28 **used to complete QAPjP and study plan submission requirements. On 12/6 and 12/10/03 FMG**
29 **agreed to prepare a table listing GLPS requirements, whether laboratory compliance was**
30 **impossible / possible at additional cost / possible at no additional cost, and whether the item is**
31 **covered by the QAPjP / in conflict with QAPjP requirements / or not addressed by QAPjP**
32 **requirements (Note: The table was not available for 1/13/04 Draft Committee discussions).**
33 **On 1/21/04 EPA reiterated that adequate quality assurance for testing aimed at sorting out**
34 **the environmental sources of PFOA and routes to human exposures is sufficient to compel**
35 **the need for full compliance with these requirements.]**
36

37 {original text}

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FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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 {12/22/03 Revised EPA placeholder text with supplemental text in red}

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

8
9 {11/24/03 FMG proposed revisions}

10 C. All testing required by this ECA must be conducted in accordance with ~~the EPA Good~~
11 ~~Laboratory Practice Standards (GLPS) found at 40 CFR part 792:~~ a Quality Assurance Project
12 Plan prepared in accordance with Appendix YYY.

13
14
15 **X. STUDY PLAN(S) AND QUALITY ASSURANCE PROJECT PLAN(S) (QAPjP)**

16
17 The Companies will submit a study plan to EPA for each test conducted pursuant to this ECA
18 prior to the initiation of testing in accordance with 40 CFR 790.62. (For this ECA, EPA will not
19 require the ~~plan(s)~~ under this Part of the ECA to be submitted “no later than 45 days prior to the
20 initiation of testing,” as specified at 40 CFR 790.62(a)). The content of the study ~~plan(s)~~ submitted to
21 EPA will comply with 40 CFR 790.62(b). This ECA and/or its appendices satisfy the applicable
22 requirements of 40 CFR 790.62(b)(2), (8), (9), and (10). A study plan may cross reference the
23 applicable provisions of the ECA and/or its appendices to satisfy these
24 requirements. Also ~~pursuant to Part IX (C) of~~ for this ECA, the Companies must submit Quality
25 Assurance Project Plan(s) (QAPjP) prepared in accordance with EPA guidance.⁴ Modifications to
26 the study plan(s) under this part of the ECA will be governed by the procedures of 40 CFR 790.62(c)
27 except that the 15 day time periods in 40 CFR 790.62(c) (2) and (3) will be 45 day time periods. All
28 study plan(s) will become part of the official record (Docket Control Number [OPPT-2003-0071]).
29

30
31 **XI. MODIFICATIONS TO THIS ENFORCEABLE CONSENT AGREEMENT**

32

4 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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FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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

3
4
5
6 **XII. FAILURE TO COMPLY WITH THE ENFORCEABLE CONSENT AGREEMENT**

7
8
9
10 ***** FOLLOW-ON DISCUSSION *****

11 [FMG views that EPA boilerplate text referring to what constitutes a failure to comply,
12 citizen's civil action, fines, and injunction to compel exceed the 40 CFR 790.60(a)(13)
13 requirements and are unnecessary. EPA could agree provided that an additional clarification
14 sentence be added (see red inserted). At the 1/13/04 meeting FMG indicated that they will
15 consider the EPA clarification sentence and discuss this at the 1/21/04 meeting]

16
17 {1/6/04 FMG proposed ~~strikeout~~ with clarifying sentence provided by EPA in red}

18 The Companies acknowledge that a violation of the requirements of this ECA will constitute a
19 "prohibited act" under section 15(1) of TSCA, 15 U.S.C. 2614(1), and will trigger all provisions
20 applicable to a section 15 violation. Further information regarding the implications of failure to
21 comply with the consent agreement is provided in 40 CFR 790.65. In addition, the Companies
22 acknowledge that noncompliance with any term of this ECA by any Company will constitute conduct
23 "in violation of this Act" under section 20(a)(1) of TSCA, 15 U.S.C. 2619(a)(1), and could result in a
24 citizen's civil action.

25
26 ~~Under the penalty provisions of section 16 of TSCA, 15 U.S.C. 2615, and the Federal Civil~~
27 ~~Penalties Inflation Adjustment Act of 1990, 28 U.S.C. 2461 note, as amended by the Debt Collection~~
28 ~~Improvement Act of 1996, 31 U.S.C. 3701 note, as implemented by 61 FR 69360 (December 31,~~
29 ~~1996), a non-complying Company could be subject to a civil penalty of up to \$27,500 per violation,~~
30 ~~with each day in violation potentially constituting a separate violation under section 15. Knowing or~~
31 ~~willful violations may lead to the imposition of criminal penalties, or a fine of not more than \$27,500 for~~
32 ~~each day of violation, or imprisonment for not more than one year, or both. In addition, EPA could~~
33 ~~enforce this ECA pursuant to section 17 of TSCA, 15 U.S.C. 2616, by seeking an injunction to~~
34 ~~compel adherence to the requirements of this ECA.~~

35
36 {Original text}

37 The Companies acknowledge that a violation of the requirements of this ECA will constitute a
38 "prohibited act" under section 15(1) of TSCA, 15 U.S.C. 2614(1), and will trigger all provisions

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

1 applicable to a section 15 violation. In addition, the Companies acknowledge that noncompliance with
2 any term of this ECA by any Company will constitute conduct "in violation of this Act" under section
3 20(a)(1) of TSCA, 15 U.S.C. 2619(a)(1), and could result in a citizen's civil action.
4

5 Under the penalty provisions of section 16 of TSCA, 15 U.S.C. 2615, and the Federal Civil
6 Penalties Inflation Adjustment Act of 1990, 28 U.S.C. 2461 note, as amended by the Debt Collection
7 Improvement Act of 1996, 31 U.S.C. 3701 note, as implemented by 61 FR 69360 (December 31,
8 1996), a non-complying Company could be subject to a civil penalty of up to \$27,500 per violation,
9 with each day in violation potentially constituting a separate violation under section 15. Knowing or
10 willful violations may lead to the imposition of criminal penalties, or a fine of not more than \$27,500 for
11 each day of violation, or imprisonment for not more than one year, or both.
12

13 In addition, EPA could enforce this ECA pursuant to section 17 of TSCA, 15 U.S.C. 2616, by seeking
14 an injunction to compel adherence to the requirements of this ECA.
15

16
17 **XIII. EPA MONITORING OF ENFORCEABLE CONSENT AGREEMENT TESTING**
18

19 EPA may conduct monitoring activities of the testing conducted under this ECA such as
20 laboratory inspections and study audits, as permitted under section 11 of TSCA, 15 U.S.C. 2610.
21

22
23 **XIV. SUBMISSIONS TO EPA AND CONFIDENTIALITY OF INFORMATION**
24

25 ***** FOLLOW-ON DISCUSSION POINT:**

26 [Summary - EPA considered the 12/10/03 FMG proposed language and provided clarification
27 text on 12/22/03. FMG rejected EPAs clarification and asked EPA to reconsider. On 1/6/04
28 EPA again rejected the FMG language. On 1/21/04 EPA proposed path forward language
29 that would be acceptable EPA and respective of the FMG position.]
30

31 {Path forward proposed by EPA for 1/21/04 discussion}

32 A. All reporting required by this ECA final reports must be submitted by the Companies to
33 EPA by the dates specified in Table 1 unless otherwise authorized by EPA pursuant to 40 CFR
34 790.68. A paper copy of a document shall be deemed submitted when it is either postmarked or
35 placed in the hands of a commercial courier service for **OVERNIGHT** delivery to EPA at the
36 appropriate address specified above in Part XIV. B. of this ECA. Hand-delivered documents are
37 deemed submitted upon receipt at the appropriate address specified in Part XIV. B. of this ECA.
38 Electronically transmitted documents are deemed delivered upon transmission and must follow the

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

1 ~~procedures for electronic submissions specified in Part XIV B. of this ECA. Under any of the above~~
2 ~~circumstances, it is the responsibility of the Companies to maintain appropriate documentation for proof~~
3 ~~of transmittal for all reporting required by this ECA.~~

4
5
6 **{Original text with 12/22/03 EPA addition in red}**

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

11
12
13 **{FMG proposed 12/10/03 additional text insert}**

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

18
19 In accordance with 40 CFR 790.62 (d), the Companies will submit interim progress reports to
20 EPA informing the Agency of any proposed changes in standards for the development of data, study
21 plans, or test schedules, and communicating with the Agency about laboratory inspections and other
22 matters affecting the progress of testing. The schedule for interim progress reports is specified in Table
23 1 of this ECA. The information required in interim progress reports is specified in Appendix E.1.

24
25 B. All documents submitted to EPA under this ECA must be identified by the Docket ID
26 Number (OPPT-2003-0071) and the name: ECA on Laboratory-Scale Incineration Testing of
27 Fluoropolymers.

28
29 Submissions made by mail should be sent to: Document Control Office (7407M), Office of
30 Pollution Prevention and Toxics (OPPT), Environmental Protection Agency, 1200 Pennsylvania
31 Avenue, NW, Washington, DC 20460-0001.

32
33 Submissions made by hand delivery or courier should be delivered to: OPPT Document
34 Control Office (DCO) in the EPA East Building, Room 6428, 1201 Constitution Avenue, NW,
35 Washington, DC and marked Attention: Docket ID Number OPPT- 2003 -0071. The DCO is open
36 from 8 a.m. to 4 p.m., Monday through Friday, excluding legal holidays. The telephone number for the
37 DCO is (202) 564-8930.

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

1 Submissions made electronically should be sent to: OPPT Document Control Office at
2 http://www.oppt.ncic@epa.gov. Attention: Docket OPPT-2003-0071. Electronic submissions do not
3 supersede the requirements of Part XIV. C. of this ECA. Electronic submissions for all reporting
4 required by this ECA must be submitted as attachments to the e-mail and must be in text-searchable,
5 PDF format. The e-mail transmitting any report required by this ECA and all electronic attachments
6 will be included as part of the submission. E-mail addresses are automatically captured by the EPA e-
7 mail system and become part of the submission that is placed in the official public docket, and will be
8 made available in the EPA electronic public docket. Upon receipt of the electronic submission, a
9 "receipt date" is entered into the metadata to signify the date the document(s) submitted by the
10 Company(ies) was received by EPA. EPA is not responsible for failure to meet a date of submission
11 requirement if the EPA fire wall rejects an electronic submission containing a virus or other adverse
12 electronic coding. It is the obligation of the submitter to confirm that: 1) electronic submissions are
13 received by EPA on the date of transmission, 2) the electronic submission and all attachments are
14 legible, and 3) the electronic submission and all attachments meet the electronic format requirements of
15 the EPA Document Control Office. Do not submit any report containing confidential business
16 information (CBI) to EPA by e-mail. For submissions containing CBI see Part XIV.D of this ECA.
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, on a
20 disk or CD ROM, of all documents submitted under this ECA (marked as CBI where appropriate and
21 in text-searchable, PDF format) will be provided to EPA. To avoid damage caused by mail scanning
22 technologies, the electronic file on disk or CD ROM must be hand delivered or sent by courier to the
23 address cited in Part XIV. A. See Part XIV. D regarding submissions containing CBI.
24

25 D. Any document submitted to EPA that contains data or information for which a
26 Signatory Company makes a claim of confidentiality (see Part XV of this ECA), must be submitted as
27 two separate versions. One version must be complete, with the information being claimed as
28 confidential marked in the manner described under 40 CFR 790.7. The other, public version must be
29 identical in all respects except that all of the information claimed as confidential shall be redacted. EPA
30 will place the public version in the Agency's docket. The complete version will be treated in
31 accordance with EPA confidentiality regulations in 40 CFR part 2 and 40 CFR 790.7.
32

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

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

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
11 ***** FOLLOW-ON DISCUSSION POINT**

12 [Summary - FMG proposed 11/24/03 additional text to clarify conditions under which EPA can
13 share a CBI Document with another government agency. EPA struck this addition on
14 12/22/03 citing laws governing such distribution adequately addressed FMG concerns. During
15 further discussion, it became clear that additional clarification could be provided to meet
16 FMG's needs. On 1/6/04 EPA excerpt text from the OPPTS CBI manual to FMG and
17 alternative language is suggested in red. On 1/13/04 discussions concluded that the EPA CBI
18 manual adequately covered in-house sharing but questions remained about external sharing.
19 EPA provided additional CBI manual excerpts to further clarify. FMG will consider this and
20 discuss at the 1/21/04 meeting.]
21

22 All results of testing conducted pursuant to this ECA will be announced to the public by EPA in
23 accordance with the procedures specified in section 4(d) of TSCA, 15 U.S.C. 2603(d). Disclosure
24 by EPA of data generated by such testing to the public or other government agencies will be governed
25 by section 14(b) of TSCA, 15 U.S.C. 2613(b), and 40 CFR part 2. The CBI version of a document
26 will only be provided to another U.S. government organization in compliance with the procedures
27 described in the OPPTS TSCA CBI Procedure Manual.
28
29

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

31 The CBI version of a document will not be provided to another government agency unless that agency
32 has certified that it affords equivalent protection.
33
34

35 **XVI. OTHER RESPONSIBILITIES OF THE COMPANIES**

36
37 ***** FOLLOW-ON DISCUSSION:** During discussions in December, FMG requested
38 that EPA reconsider designating the chemicals listed in Appendix A.1 as the ECA test

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

1 substances in lieu of identifying the composites as the test substances. On 1/6/04 EPA
2 indicated that it would accept FMG's request. Since one composite contained only one
3 CAS number, that composite would be subject to 12(b) reporting. On 1/13/04 EPA
4 provided language to support this change as indicated in red and strikeout below.
5 FMG rejected this language and sought the following additional changes from EPA: 1)
6 delete the name and CAS # for PTFE and provide only a cross reference to Part II of
7 the ECA for test substances subject to 12(b), 2) delete the dry non-melt resin
8 composite as a test substance in Part II and Appendices. On 1/21/04 EPA indicated
9 that it does not agree to these changes for reasons stated above and to maintain
10 clarity in the ECA document.

11
12
13 {EPA revised text of 1/13/04}

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

22
23 B. If any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA become
24 subject to a rule promulgated under TSCA section 5(a)(2), 15 U.S.C. 2604(a)(2), governing significant
25 new uses of any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA, then the
26 Companies will be subject to the data submission requirements imposed by section 5(b)(1)(A) of
27 TSCA, 15 U.S.C. 2604(b)(1)(A), as if the testing under this ECA had been required by a TSCA
28 section 4 test rule.

29
30
31 **XVII. SEVERABILITY OF ENFORCEABLE CONSENT AGREEMENT PROVISIONS**

32
33 In the event that one or more provisions of this ECA are determined by a court decision to be
34 unenforceable, the remaining provisions of this ECA will not be presumed to be valid, and EPA will
35 either initiate a rulemaking proceeding to require testing or publish in the Federal Register the reasons
36 for not initiating such a proceeding.

DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE

1 **XVIII. FINAL AGENCY ACTION**

2
3 For purposes of 5 U.S.C. 704, publication of the FR notice announcing the issuance of the
4 Order incorporating this ECA constitutes final agency action..

5
6
7 **XIX. PUBLIC RECORD**

8
9 EPA has established a public record which will contain this ECA, the Order that incorporates
10 this ECA, the Federal Register notice announcing issuance of the Order incorporating this ECA, and
11 any and all relevant information, subject to the confidentiality provisions of section 14(b) of TSCA and
12 40 CFR part 2. The official record for this ECA, including the public version, which does not include
13 any information claimed as CBI, has been established under Docket Control Number OPPT-2003-
14 0071.

15
16 An electronic version of the public docket is available through EPA's electronic public docket
17 system, EPA Dockets. EPA Dockets may be accessed at <http://www.epa.gov/edocket/> to access the
18 index listing of the contents of the official public docket, and to access those documents in the public
19 docket that are available electronically. Although not all docket materials may be available
20 electronically, (for example the materials in the original dockets for this action, [AR-226 and OPPTS-
21 2003-0012], or materials under copyright), can be access any of the publicly available docket materials
22 through the EPA Docket Center, Rm. B102-Reading Room, EPA West, 1301 Constitution Ave.,
23 NW., Washington, DC. For materials available in the electronic docket, once in the system, select
24 "search," then key in the appropriate Docket ID number (OPPT-2003-0071).

25
26
27 **XX. EFFECTIVENESS**

28
29 This ECA may be signed in separate counterparts. This ECA will not be effective unless signed
30 by each of the Companies and by EPA. This ECA will take effect on the date of publication of the
31 Federal Register notice announcing the issuance of the Order that incorporates this ECA.

32
33
34 **XXI. RIGHTS OF THE COMPANIES**

35
36 By signing this ECA, the Companies waive their right to challenge EPA's authority to assess
37 penalties for violations of the terms of this ECA. This waiver does not affect any other rights that the
38 Companies may have under TSCA, including the right to dispute the amount of any penalty or to

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1 dispute factually whether a violation of the terms of this ECA has occurred, or to seek judicial review of
2 any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer
3 chemicals listed in Appendix A.1 to this ECA.
4

5
6 **XXII. RESERVATION OF RIGHTS BY COMPANIES**
7

8 By signing this ECA, the Companies are not admitting that the requirements of TSCA Section 4
9 have been satisfied for promulgating a test rule to generate the data required by this ECA.
10

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

1 **XXIII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR**

2
3 The Principal Test Sponsor is:

4
5 Fluoropolymer Manufacturers Group

6 **[? Name of technical contact person ?]**

7 **[? ADDRESS ?]**

8 **[? Phone Number ?]**

9
10
11 The Companies subject to this ECA are:

12
13
14 Asahi Glass Fluoropolymers USA, Inc.

15 **[? ADDRESS ?]**

16
17
18 Daikin America, Inc.

19 **[? ADDRESS ?]**

20
21
22 Dyneon, LLC

23 **[? ADDRESS ?]**

24
25
26 E.I. du Pont de Nemours and Company

27 **[? ADDRESS ?]**

28

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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

2
3 **XXIV. SIGNATURE**

4 **TEST SPONSOR**

5 **Asahi Glass Fluoropolymers USA, Inc.¹**

6

7 **ECA Subject Chemicals for**

8 **Asahi Glass Fluoropolymers USA, Inc.**

9 CAS No.	Chemical Name	Composite(s)
10		
11		
12		
13		
14		

15

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

17

18 Name: _____

19 Title: _____

20 Address: _____

21 Phone Number: _____

22

23

24 Date: _____

25 [? NAME ?]

26 [? TITLE ? e.g., Senior Vice President]

27 Asahi Glass Fluoropolymers USA, Inc.

28 [? ADDRESS ?]

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

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1 Special Page Header: ECA Copy # 4

Daikin America, Inc.

2
3 **XXIV. SIGNATURE**

4 **TEST SPONSOR**
5 **Daikin America, Inc.¹**

6

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

7
8
9
10
11
12
13
14

15
16 Company technical contact person for handling correspondence marked as "Confidential"

17
18 Name: _____
19 Title: _____
20 Address: _____
21 Phone Number: _____

22
23
24
25 Date: _____

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

26
27
28
29

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

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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

XXIV. SIGNATURE

TEST SPONSOR

E.I. du Pont de Nemours and Company¹

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

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

Name: _____
Title: _____
Address: _____
Phone Number: _____

Date: _____

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

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

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FLUOROPOLYMER ECA DRAFTING COMMITTEE**

1 Special Page Header:

2 ECA Copy # 1 EPA PUBLIC VERSION
3 CONTAINS NO CONFIDENTIAL BUSINESS INFORMATION
4

5 Special Page Header:

6 ECA Copy # 2 EPA CBI VERSION
7 CONTAINS CONFIDENTIAL BUSINESS INFORMATION
8

9
10 **XXIV. SIGNATURE**

11
12
13
14
15
16
17
18 **UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**
19
20
21
22
23
24
25
26
27

28
29 Date: _____

30 Stephen L. Johnson
31 Assistant Administrator
32 Office of Prevention, Pesticides, and Toxic Substances
33

34
35 Address:

36 U.S. Environmental Protection Agency
37 Office of Prevention, Pesticides, and Toxic Substances
38 Ariel Rios Building
39 1200 Pennsylvania Avenue, N.W.
40 Washington, DC 20460

**DRAFT DOCUMENT - DO NOT CITE OR QUOTE - January 21, 2004
FLUOROPOLYMER ECA DRAFTING COMMITTEE**

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

Phase I PFOA Transport Testing	Test Standard	Deadline for Final Report (Months)
Study Plan(s)	40 CFR 790.62 (b)	?TBD ?
QAPP submission	see Guidance Manual	?TBD ?
Quantitative PFOA transport analysis ²	See appendix C.1 as annotated in appendix D.?)	?TBD "4" ? ³

13

¹ 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 XIV. of this ECA).

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

³ In the event that the transport efficiency of PFOA or of total fluorine (as determined by the formulas in Appendix C.1) is less than 70% and the Technical Consultation concludes that testing can not proceed to Phase II, then the Companies 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.

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Phase II Fluoropolymer Incineration Testing ⁴	Test Standard	Deadline for Final Report (Months) ⁵
Study Plan(s)	40 CFR 790.62 (b)	?TBD?
QAP/P submission	see Guidance Manual	?TBD?
Elemental analysis	See Appendix C.2.1 of the ECA	?TBD "4"?
Combustion stoichiometry	See Appendix C.2.2 of the ECA	?TBD "4"?
Thermogravimetric analysis	ASTM E 1868-02 (as modified by Appendix B.1 of the ECA)	?TBD "6"?
Laboratory-scale combustion testing	See Appendix C.2.4 of the ECA (as annotated by Appendix D.1, D.2 and D.3 of the ECA)	?TBD "18"?
Release assessment report ⁶	(see Appendix E.2 of the ECA)	?TBD "20"?

⁴ Phase II testing will begin ??TBD??

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

⁶ 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.2)) resulting from the combustion testing for any or all of the fluoropolymer composites to be tested under this ECA, then the Companies will prepare a release assessment report to put into perspective the relevance of the laboratory-scale incineration testing data with respect to municipal and/or medical incineration operations in the United States (see Appendix E.2 to this ECA).

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Note to the Drafting Committee:

The following pages provide a compilation of most recent (as of 1/15/04) documents available to form a draft of the various appendices that will be attached to the draft ECA document for the Laboratory-Scale Incineration Testing of Fluoropolymers. An earlier date in the top right hand margin of the appendix indicates that it is a prior draft version and does not necessarily reflect changes incorporated from the last meeting. This will be updated as FMG provides updates of their contributions to these appendices.

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APPENDIX A

TEST SUBSTANCES

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

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APPENDIX A.1

LIST OF CHEMICAL COMPONENTS OF THE COMPOSITES
- SUBJECT TO THIS ECA¹

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

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

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

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

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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, 1,1,1,2,2,3,3,3-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
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 <u>Accession No. ????</u>	<u>??generic name??</u>

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APPENDIX A.2

RATIONALE FOR SELECTING COMPOSITES TO BE TESTED

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APPENDIX A.2
2. Selection

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

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

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

- Dry melt resins
 1. FEP, PFA, THV, ETFE, HTE
- Dry non-melt resins and gums
 2. PTFE resin
 3. Fluoroelastomer gums
- Aqueous dispersions
 4. PTFE, FEP, PFA, THV

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

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APPENDIX A.3

COMPOSITION OF COMPOSITES TO BE TESTED¹

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

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1 APPENDIX A
 2 APPENDIX A.3
 3 1. Identification

4 The four composite test substances for this test program
 5 will be prepared from the fluoropolymers identified in the
 6 March 14, 2003 Letter of Intent (LOI) submitted by the
 7 Society of the Plastics Industry on behalf of the four LOI
 8 signatories (as corrected). The specific fluoropolymer
 9 types (with CAS numbers and associated monomers) going into
 10 each of the four composite test substances (grouped as
 11 shown) are presented in Table A-1 below. Each
 12 fluoropolymer used in each relevant test substance
 13 composite will have been made using APFO.
 14
 15

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

Test Substance	Fluoropolymer Type	CAS Number	Associated Monomers
Composite 1 - PTFE resin (dry non-melt)	PTFE	9002-84-0	TFE
Composite 2 - Dry melt resins	FEP	25067-11-2	TFE, HFP
	PFA	26655-00-5	TFE, PPVE
		31784-04-0	TFE, PEVE
	THV	25190-89-0	TFE, HFP, VDF
	ETFE	68258-85-5	TFE, E
Composite 3 - Fluoroelastomers (dry non-melt)	HTE	35560-16-8	TFE, HFP, E
	Fluoroelastomer Copolymers	9011-17-0	VDF, HFP
		25190-89-0	TFE, HFP, VDF
	Fluoroelastomer Terpolymers	54675-89-7, 27029-05-6	TFE, VDF, P TFE, P
		Perfluoroelastomers	26425-79-6
	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 Confidential business information (CBI) regarding the
2 chemical identity of Low temperature elastomers has
3 previously been submitted to EPA under separate cover.
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APPENDIX A.4

PREPARATION OF COMPOSITES TO BE TESTED¹

¹ 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 XIV of this ECA regarding confidentiality of information).

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31 APPENDIX A.4
32 3. Preparation of Fluoropolymer Composites

33
34 3.1 Approach

35
36 A composite mixture of representative fluoropolymers, as
37 solids, will be prepared for each of the four test
38 substance composites identified in Table A-1.

39
40 The polymer samples will be first quality product polymer,
41 substantially free of inorganic constituents. Each sample
42 will be from a representative grade for each applicable
43 fluoropolymer type from each applicable company.

44
45 A hypothetical example for Composite 2 in Table A-2 below
46 shows how the composites will be assembled. In this

1 example with 4 types across 4 companies, there are 11 x's.
2 Hence, composite 2 would be made up of 11 equal proportions
3 of the materials indicated with an x.

4
5 Table A-2. Example for Compositing Across Companies & Types

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

6
7 For Composite 1 - PTFE resin (where there is a single
8 fluoropolymer type), a representative sample of PTFE resin
9 from each company producing PTFE resin will be mixed
10 together in equal proportions across applicable companies
11 to form the Composite 1 - PTFE resin.

12 3.2 Preparation

13
14 Representative samples of each component from each
15 applicable company for each composite will be sent to the
16 laboratory(ies) in packaging customarily used for product
17 sample packaging or in polyethylene, polypropylene, or
18 glass containers.

19
20 Each composite will be prepared under laboratory conditions
21 designed to prevent cross-contamination and designed to
22 assure solids temperatures less than or equal to 60 °C.

23
24 Following preparation of each composite, the composite will
25 be placed in a polyethylene, polypropylene, or glass
26 container.

27 3.2.1 Composite 1

28
29 PTFE resin is available in powder form. Equal weights of
30 PTFE resin powder samples across applicable companies will
31 be mixed together in dry form to yield Composite 1.

32 3.2.2 Composite 2

33
34 PEP, PVA, THV, PTFE, and RTE dry melt resins are available
35 in pellet form. Each component of Composite 2 will be
36 size-reduced (e.g., ground) to produce powder. Equal
37 weights of the powder form of each component following the
38

1 approach in the example for Composite 2 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 *(text on size reduction blank under further development)*

9
10 3.2.3 Composite 3

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

15
16 a) Equal weights of each component (following the approach
17 in example for Composite 2 in Section 3.1) will be mixed
18 on a rubber mill to produce a homogenous slab of preset
19 thickness to yield Composite 3.

20
21 Or

22
23 b) Each component of Composite 3 will be cryogenically
24 cooled (to make the elastomers brittle) and size-reduced
25 (e.g., ground) to produce powder. Equal weights of the
26 powder form of each component (following the approach in
27 the example for Composite 2 in Section 3.1) will be mixed
28 together in dry form to yield Composite 3.

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

34
35 *(text on size reduction blank under further development)*

36
37 3.2.4 Composite 4

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

43
44 a. Equal weights (on a dry solids basis) of each component
45 in aqueous dispersion form (following the approach in
46 example for Composite 2 in Section 3.1, will be mixed
47 together in liquid form. Solids will be separated from

1 the resulting liquid composite to yield low water content
2 (i.e., drip free) fine solids.

3
4 Or

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

12
13 3.3 Verification

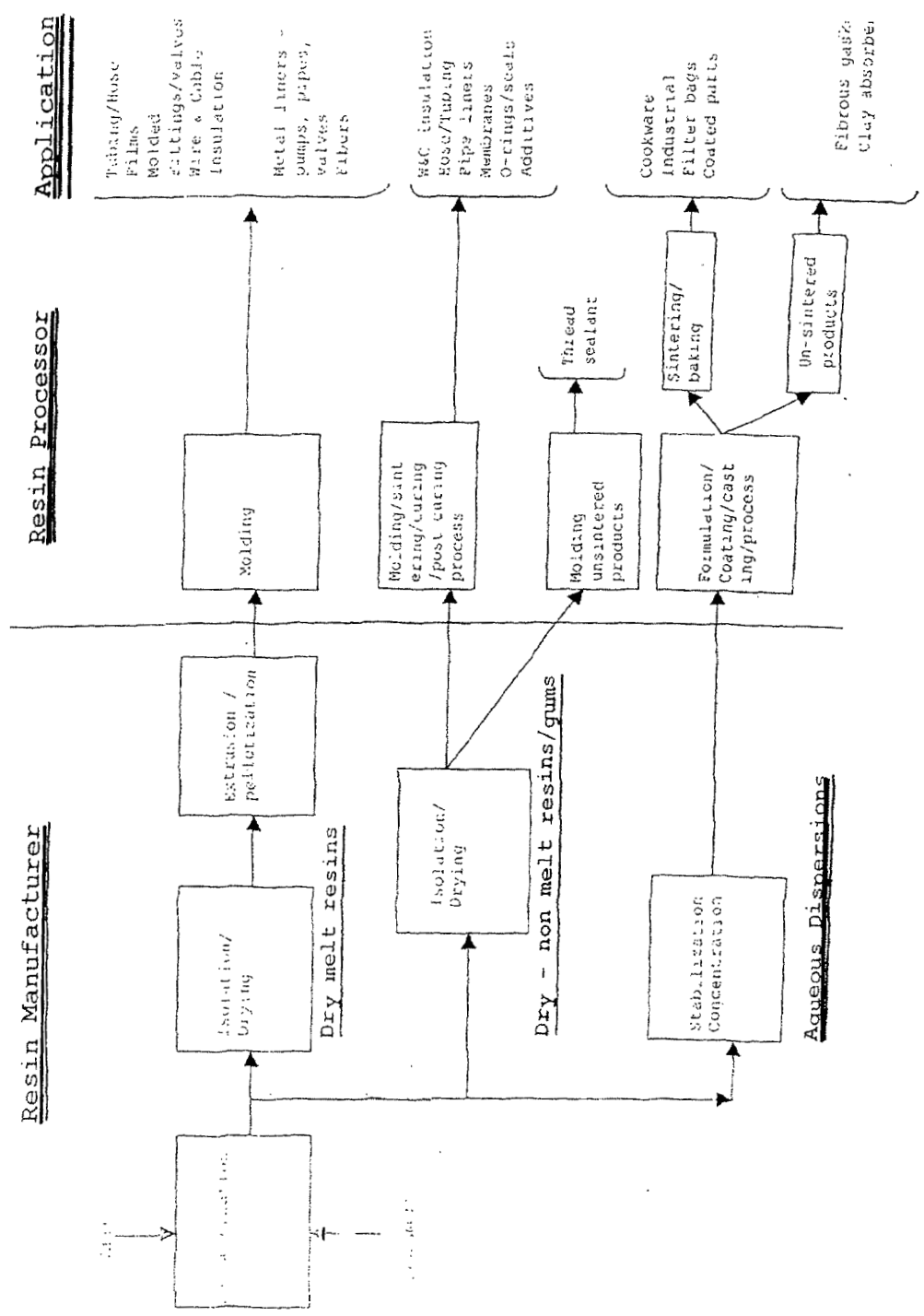
14
15 In order to assure that composite samples in this testing
16 program have been made up of clearly identified materials,
17 the preparation of the composites will include formal Chain
18 of Custody procedures. A chain of custody form will be
19 included with each component material going into the
20 composite to show the identity of the component material
21 and each transfer of custody from its point of origination
22 to preparation of the composite. For documentation, the
23 laboratory preparing a given composite will generate a
24 report to be submitted to EPA as CBI along with a sanitized
25 version for the public record from which CBI has been
26 removed.

27
28 Once prepared, each composite will be accompanied by a new
29 chain of custody until it reaches the incineration testing
30 facility.

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Figure A-1. Fluoropolymer Industry Overview



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APPENDIX B

TEST STANDARDS

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

APPENDIX B.1
GUIDELINE FOR THERMOGRAVIMETRIC ANALYSIS

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

Section	Exception/Modification
2.1	<ul style="list-style-type: none">Standard practices at the University of Dayton Research Institute (UDRI) may be used as references throughout the standard in place of the ASTM standards noted in this section.
4.1	<ul style="list-style-type: none">The loss-on-drying (LOD) value specified in the second through fifth sentences of this section will not be recorded.
7.1.3	<ul style="list-style-type: none">The programming rate of the furnace will be set at 10 to 25°C/min, rather than 5°C/min. Pursuant to section 11.6, the temperature program rate will be documented in the report.The isothermal temperature within the range of 25 to 1000°C will be maintained ±3°C, rather than ±2°C.
7.1.4	<ul style="list-style-type: none">The specimen atmosphere control system will be capable of supplying dry air in addition to "inert dry gas (usually purified grade nitrogen)".
7.1.7	<ul style="list-style-type: none">The temperature program rate will be set at 10 to 25°C/min, rather than 5°C/min. Pursuant to section 11.6, the temperature program rate will be documented in the report.The temperature program rate will be controlled to within the range of ±3°C/min, rather than ±0.1°C/min.Within the range of 25 to 1000°C, the isothermal temperature will be maintained within ±3°C, rather than ±2°C.
11.4	<ul style="list-style-type: none">The mass of the test specimen noted in the first sentence of this section will be 0.95-005 to 5 mg, rather than 10±1 mg (i.e., 9 to 11 mg).
11.6	<ul style="list-style-type: none">The test specimen heating rate will be set at 10 to 25°C/min, rather than 5°C/min noted in the first sentence of this section. Pursuant to section 11.6, the temperature program rate will be documented in the report.
11.9	<ul style="list-style-type: none">Termination criteria will follow Test Method A as

B.1-1

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

Reference

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

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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 Study Reporting**

1 **APPENDIX C.1**
2 **PFOA TRANSPORT TESTING**

3
4 C.1.1 Significance

5
6 Testing will be performed to verify that potential PFOA
7 emissions from the combustion testing described in Appendix
8 C.2 can be quantitatively transported from the high
9 temperature reactor into the exhaust gas sampling apparatus
10 (aqueous solution bubblers).

11
12 Acceptable PFOA transport will be demonstrated if the
13 transport efficiency (as computed in one or more of the
14 formulas below) is greater than or equal to 70%.

15
16 C.1.2 Experimental Plan

17
18 C.1.2.1 Base Plan

19
20 Transport of PFOA across the laboratory-scale thermal
21 reactor system described in Appendix C.2.4 and into the
22 exhaust gas bubblers described in Appendix D.1 will be
23 quantitatively determined as an indication of transport
24 from the high temperature reactor into the bubblers.

25
26 A PFOA standard of known purity greater than or equal to
27 97% will be gasified at 150 to 250 °C (based on
28 thermogravimetric analysis of PFOA) with transfer line and
29 reactor temperatures 0 to 100 °C higher than the
30 gasification temperature.

31
32 Three replicate transport efficiency test runs will be
33 conducted. A minimum of one blank run will be conducted
34 prior to each transport efficiency test run.

35
36 The sample size of the PFOA standard to be gasified will be
37 less than 5 mg. The reactor exhaust gas will be collected
38 into bubbler aqueous solution as described in Appendix D.1
39 (including an HPLC water rinse of the flexible tubing [used
40 to connect the thermal reactor system and the bubbler
41 assembly] into the aqueous solution composite), which will
42 be analyzed for PFOA as described in Appendix D.2. In
43 order to provide a second way of demonstrating quantitative
44 transport, this aqueous solution composite will also be
45 analyzed for total fluorine as described in Appendix D.3.
46 (Testing for total fluorine is included due to possibility
47 of thermal degradation of PFOA under transport test

C.1-1

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1 conditions.) Therefore, for this transport testing the
2 sample size of PFOA standard will be sufficiently high to
3 assure that the total fluorine input to the thermal reactor
4 system will be greater than 140% of the mass corresponding
5 to the limit of quantitation (LOQ) for total fluorine in
6 the aqueous solution composite. (The LOQ for total
7 fluorine in aqueous solution is much higher than the LOQ
8 for PFOA in aqueous solution.)
9

10 The amount of PFOA and total fluorine in the thermal
11 reactor system exhaust gas will be determined via analysis
12 of the aqueous solution composite as noted above.
13

14 The amount of PFOA fed to the thermal reactor system will
15 be known based on measurement prior to gasification and
16 will be verified by weighing the pyroprobe insert cartridge
17 before and after each test run. The amount of fluorine fed
18 to the system will be calculated from the amount of PFOA
19 fed, the known purity of the PFOA, and the known fluorine
20 fraction of the PFOA standard.
21

22 PFOA transport efficiency (TE) as a percentage will be
23 computed as follows:
24

$$25 \quad \% \text{ PFOA TE} = \frac{\text{mass of PFOA in aqueous solution composite}}{\text{mass of PFOA fed to thermal reactor system}} * 100 \quad (1)$$

26

27
28 Total fluorine (TF) transport efficiency as a percentage
29 will be computed as follows:
30

$$31 \quad \% \text{ Total F TE} = \frac{\text{mass of total F in aqueous solution composite}}{\text{mass of total F fed to thermal reactor system}} * 100 \quad (2)$$

32

33 34 C.1.2.2 Contingent Testing

35
36 If the transport efficiencies for both PFOA (equation 1)
37 and total fluorine (equation 2) are less than or equal to
38 70%, then additional work will be performed. ~~as described~~
39 ~~in Section C.1.2.2.~~
40

41 ~~C.1.2.2 Contingent Testing~~

42
43 ~~As indicated by Section C.1.2.1, This~~ additional work will
44 be performed, ~~as necessary,~~ in a step-wise fashion to
45 determine if consideration of one or more of the following
46 procedural revisions enables achievement of 70% transport
47 efficiency as follows:
48

C.1-2

1 Step 1. The flexible tubing between the thermal reactor
2 system and the bubbler assembly from the experiment
3 described in Section C.1.2.1 would be
4 quantitatively rinsed with methanol. This methanol
5 rinsate would be analyzed for PFOA (as described in
6 Appendix D.2) and/or for total fluorine (as
7 described in Appendix D.3). Revised transport
8 efficiency (TE) as a percentage for PFOA (equation
9 3) and/or total fluorine (equation 4) would be
10 computed by including the mass of analyte in the
11 methanol rinse in the numerator as follows:

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

17 where $\text{mass}_{\text{PFOA out}} =$ mass of PFOA in bubbler
18 aqueous solution composite
19 + mass of PFOA in methanol
20 rinse

21 and $\text{mass}_{\text{PFOA in}} =$ mass of PFOA fed to thermal
22 reactor system

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

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

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

39 Step 2 (if necessary) ~~Reagent(s) would be added to the~~
40 ~~bubbler aqueous solution, and the~~ The
41 experiment described in Section C.1.2.1
42 would be repeated with reagent(s) (e.g.
43 NaOH) added to the bubbler aqueous
44 solution to determine if reagent
45 addition enhances analyte absorption,
46 thereby improving transport efficiency.
47 Transport efficiency would be
48 calculated using equation (1) and/or
49 (2) above. The impact of reagent
50 addition on LOQ for PFOA analysis
51 described in Appendix D.2 would be

1. **APPENDIX C.2**
2. **INCINERATION TESTING**

3
4. **C.2.1 Elemental Analysis**

5
6. **C.2.1.1 Introduction**

7
8 Elemental analysis as described in Section C.2.1 will be
9 performed to aid in preparation for combustion testing
10 described in Section C.2.4.

11
12 As Kissa (1998) points out, technique strongly affects
13 analytical results for fluorinated organic compounds such
14 as fluorinated surfactants and fluorinated polymers due to
15 the strength of the carbon-fluorine bond:

16
17 Fluorine in organic compounds is usually determined by
18 converting organic fluorine to an inorganic fluoride.
19 Various combustion methods are routinely used for this
20 purpose. However, the carbon-fluorine bond is
21 exceptionally strong, and extremely vigorous conditions are
22 needed for a quantitative mineralization. Conventional
23 combustion conditions used for the determination of carbon
24 and hydrogen in nonfluorinated organic compounds are not
25 adequate for a quantitative analysis of fluorinated
26 surfactants.

27
28 Therefore, total fluorine analysis will be performed using
29 "extremely vigorous conditions" as described in Section
30 C.2.1.2, and the commercially available conventional
31 technique used for empirical determination of carbon and
32 hydrogen content (described in Section C.2.1.3) will
33 provide estimated values.

34
35. **C.2.1.2 Total Fluorine**

36
37 Each test substance composite will be characterized via
38 analysis of total fluorine content.

39
40 Based on manufacturing process knowledge, the levels of
41 total fluorine in the components of test substance
42 composites are orders of magnitude higher than the
43 potential trace level of inorganic fluoride in these
44 materials. Therefore, for this test program, the total
45 organic fluorine value for each test substance composite
46 will be considered to be the same as the total fluorine
47 value.

48
49 Total fluorine content will be measured via the Wickbold

C.2-1

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1 Torch method; see Appendix D.3.

2

3 C.2.1.3 Carbon and Hydrogen

4

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

12

13 C.2.1.3.1 Theoretical Determination

14

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

19

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

24

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

25

26 C.2.1.3.2 Empirical Determination

27

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

32

33 As noted in Section C.2.1.1, empirical determination of
34 carbon in test substance composites via commercially
35 available conventional techniques is expected to
36 underestimate the carbon content of the sample due to the
37 strength of the carbon-fluorine bond. Similarly, empirical
38 determination of hydrogen in test substance composites via
39 commercially available conventional techniques is expected
40 to overestimate the hydrogen content of the sample.

41

42 The carbon content of the sample is measured by determining
43 the carbon dioxide (CO_2) generated by the oxidation of the

C.2-2

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1 sample. This oxidation may be accomplished by high
2 temperature combustion, catalytic combustion, or wet
3 chemical oxidation. The CO₂ is measured directly by an
4 infrared detector or a thermal conductivity detector, via
5 absorption into a suitable solution (e.g., potassium
6 hydroxide) and gravimetric determination, or by conversion
7 to methane for measurement via a flame ionization detector.

8
9 The hydrogen content of the sample can be determined by
10 difference with knowledge of the fluorine content and
11 carbon content of the sample where the moisture content and
12 chlorine content of the sample are negligible or known.
13 Alternatively, the hydrogen content of the sample is
14 measured by determining the water generated by high
15 temperature combustion of the sample. Measurement of water
16 in the combustion gas for this analysis may be accomplished
17 by techniques such as use of an infrared detector or
18 absorption on a desiccant with gravimetric determination.
19 With empirical hydrogen determination, it is important to
20 correct for the water in the combustion gas attributable to
21 the moisture content in the sample to obtain the hydrogen
22 content of the sample; see Section C.2.1.4.

23
24 Manufacturing process knowledge of the polymers will be
25 used to review the elemental analysis results and to form
26 the basis for interpreting non-detects. For example, if
27 the hydrogen analytical result for a perfluorinated polymer
28 is less than a quantitation limit of 0.1%, then the
29 analytical result will be replaced with 0.

30
31 C.2.1.4 Moisture

32
33 Where preparation (as described in Appendix A.4) for a
34 given test substance composite has involved dewatering, the
35 moisture (or solids) content of each such test substance
36 composite will be determined in order to provide a dry
37 basis for calculations as needed.

38
39 Moisture is determined by measuring the loss of weight of
40 the sample when heated under controlled conditions. A
41 representative sample is weighed and placed in a crucible
42 (or dish) and evaporated to dryness in an air or nitrogen
43 atmosphere at a defined temperature setpoint (e.g., 103 °C
44 to 105 °C) in the range of 100 °C to 125 °C.

45
46 The moisture value is calculated as the loss in weight
47 (difference between the starting weight of sample and the

C.2-3

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1 final weight of sample) divided by the starting weight of
2 sample. Similarly, a solids value can be calculated as the
3 final weight of sample divided by the starting weight of
4 sample.

5 6 **C.2.2 Combustion Stoichiometry**

7
8 Combustion stoichiometry calculations as described in
9 Section C.2.2 will be performed to aid in preparation for
10 combustion testing described in Section C.2.4

11
12 First, the weight percent values from Section C.2.1 are
13 converted to molar quantities on a dry basis.

14
15 Second, based on Chapter 3 of *Combustion Fundamentals for*
16 *Waste Incineration* (American Society of Mechanical
17 Engineers, 1974), the reaction products for these molar
18 quantities are calculated assuming complete combustion with
19 the following rules:

20
21 a) All carbon (C) in feed converts to carbon dioxide (CO₂)
22 $C + O_2 \rightarrow CO_2$

23
24 b) All sulfur (S) in feed converts to sulfur dioxide (SO₂)
25 $S + O_2 \rightarrow SO_2$

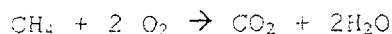
26
27 c) The halogens (Cl, F) in feed convert to hydrogen halides
28 $H_2 + Cl_2 \rightarrow 2HCl$
29 $H_2 + F_2 \rightarrow 2HF$

30
31 d) Hydrogen (H) present in feed in excess of that
32 required to yield products in item c) above will be
33 converted to water
34 $2H_2 + O_2 \rightarrow 2H_2O$

35
36 e) Nitrogen (N) from feed or air is emitted as molecular
37 nitrogen
38 $N_2 \rightarrow N_2$

39
40 Third, with these rules, the balanced chemical reaction for
41 combustion of a compound can be written.

42
43 For example, the resulting reaction equation for a
44 hydrocarbon like methane (CH₄) is



47

C.2-4

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

7
8 These calculations provide the theoretical amount of oxygen
9 needed for the overall combustion reaction for the feed
10 based on the available information used in the
11 calculations. The initial estimate for the amount of
12 oxygen to be used in combustion testing will be determined
13 from this theoretical amount with adjustments for target
14 oxygen level in thermal reactor system exhaust gas. The
15 actual amount of oxygen to be used in combustion testing
16 will be based oxygen monitoring described in Section C.2.4.

17
18 These stoichiometric calculations will also be used as
19 needed to initially estimate and adjust experimental
20 conditions for combustion testing in Section C.2.4.

21 **C.2.3 Thermogravimetric Analysis**

22
23
24 Thermogravimetric analysis (TGA) will be conducted to
25 determine the temperature range required for gasification
26 of each test substance composite. TGA will be conducted in
27 flowing air from room temperature to 1000°C as described in
28 Appendix B.1.

29
30 The TGA weight-loss profile for each test substance
31 composite will be evaluated to determine the temperature at
32 which the weight loss reaches a final asymptote across the
33 temperature range investigated. This temperature
34 corresponds to the point at which no further gasification
35 (under test conditions) occurs for the material and will be
36 considered the temperature for complete gasification of the
37 material.

38 39 **C.2.4 Combustion Testing**

40 41 **C.2.4.1 Test Objective**

42
43 The objective of the testing program described in Appendix
44 C.2 is to assess the potential for waste incineration of
45 each test substance composite to emit PFOA, based on
46 quantitative determination of potential exhaust gas levels
47 of PFOA from laboratory-scale combustion testing under

C.2-5

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1 conditions representative of typical municipal waste
2 combustor operations in the U.S.

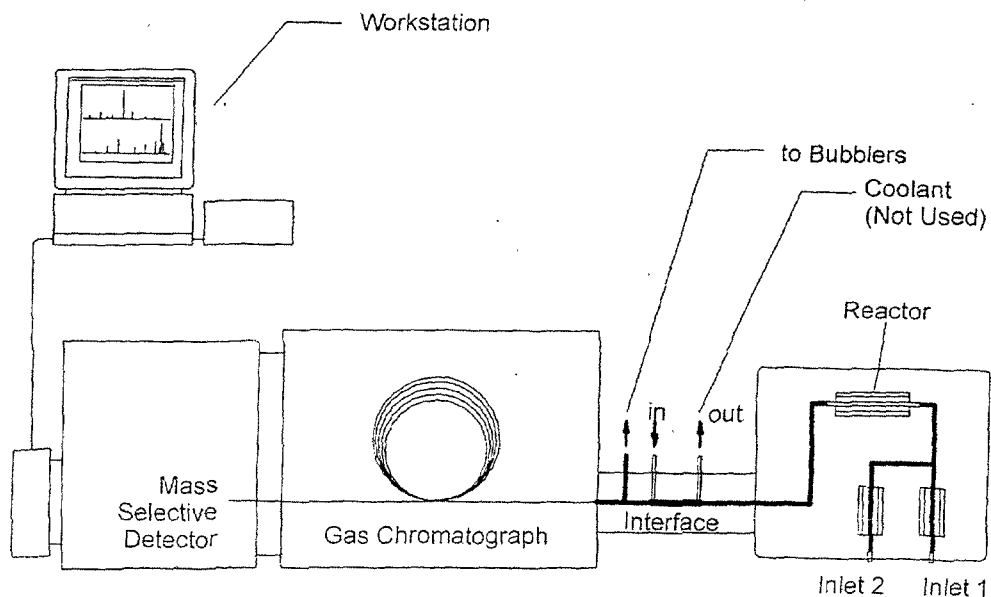
3
4 C.2.4.2 Experimental Apparatus

5
6 Combustion testing will make use of the Advanced Thermal
7 Reactor System (ATRS) at the University of Dayton Research
8 Institute (UDRI). The ATRS is a laboratory-scale, non-
9 flame, batch-charged, continuous flow thermal reactor
10 system. The use of this non-flame thermal reactor system
11 gives a conservative representation of full-scale waste
12 incineration prior to air pollution controls.

13
14 In the ATRS, the test sample is gasified and transported to
15 a high temperature reactor. In the high temperature
16 reactor, the sample vapors are subjected to controlled
17 conditions for residence time and temperature. As
18 described in Sections C.2.4.5 and C.2.4.6, combustion
19 products will be monitored or collected for quantitative
20 analysis.

21
22 A schematic of the ATRS as configured for this test program
23 is shown in Figure C.2-1.

24
25 **Figure C.2-1. Schematic of ATRS for this Test Program**



26
27 The ATRS consists of a reactor assembly and in-line gas
28 chromatograph/detector system connected via an interface.

C.2-6

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

4
5 Sample introduction for solid materials (Inlet 1) employs a
6 pyroprobe, a device designed to gasify samples by heating
7 them at a fixed rate. The main gas flow will also be fed
8 via Inlet 1, and Inlet 2 will be used to feed supplemental
9 flow.

10
11 During combustion tests, the transfer line between the
12 pyroprobe and the reactor is heated and maintained above
13 200 °C. The reactor is housed within its own small tube
14 furnace and may be independently heated to as high as 1100
15 °C. (Actual conditions for this test program are presented
16 in Section C.2.4.3.) The transfer line from the reactor to
17 the interface is heat traced to greater than 200 °C to
18 prevent cool regions where reactor products could otherwise
19 be lost through condensation.

20
21 The interface routes the combustion exhaust gas to the in-
22 line gas chromatograph (GC) and mass selective detector
23 (MSD) or to sample collection for off-line analysis. For
24 combustion testing in this test program, the interface will
25 also be maintained above 200 °C. Exhaust gas monitoring for
26 this program is described in Section C.2.4.5.

27
28 C.2.4.3 Combustion Test Experimental Conditions

29
30 Each test substance composite will be subjected to
31 laboratory-scale incineration using the experimental
32 apparatus described in Section C.2.4.2.

33
34 C.2.4.3.1 Combustion Air

35
36 Synthetic air (mixture of 21% oxygen and 79% nitrogen) will
37 be used in place of compressed air to prevent potential
38 interference in the experimental system due to background
39 levels of CO₂ in compressed air.

40
41 C.2.4.3.2 Fuel

42
43 Methanol will be used, as needed, as a supplemental fuel to
44 ensure the presence of sufficient hydrogen to convert
45 fluorine to hydrogen fluoride (HF) and chlorine to hydrogen
46 chloride (HCl).

47

1 As noted in *Municipal Solid Waste in the United States:*
2 *2000 Facts and Figures* (EPA, 2002), paper and paper
3 products (made from wood) make up the largest component of
4 municipal solid waste (MSW). The sum of paper and paper
5 products with wood in MSW makes up over 30% of MSW.

6
7 During the 19th century, methanol was produced from wood and
8 was known as wood alcohol. Therefore, methanol can be used
9 in this experimental program as a surrogate for the paper
10 and wood fraction of MSW.

11
12 C.2.4.3.3 Operating Conditions

13
14 The target operating conditions for the high temperature
15 reactor during the combustion tests for each test substance
16 composite identified in Appendix A.3 are presented in Table
17 C.2-1.

18
19 **Table C.2-1. Combustion Test Target Operating Conditions**

Temperature	1000 °C
Residence Time	2 sec
O ₂ concentration in exhaust gas	10%
H ₂ O concentration in exhaust gas	14%
Number of replicate runs	3

20
21 These conditions are representative of typical furnace
22 operating conditions of municipal waste combustors (MWCs)
23 and of typical secondary chamber operating temperatures for
24 medical waste incinerators in the U.S. See Appendix D.4
25 for supporting information.

26
27 Temperature and residence time values in Table C.2-2 will
28 be fixed setpoints for the experiment. The temperature of
29 the high temperature reactor will be controlled within ± 10
30 °C to assure isothermal operation.

31
32 The ATRS sample size for the test substance composites in
33 this testing program will be a measured amount less than 5
34 mg. The actual sample size, gasification rate (determined
35 from TGA), air supply, and fuel supply will be adjusted to
36 assure that the oxygen level in the exhaust will be greater
37 than or equal to the concentration in Table C.2-1
38 throughout each test to be representative of typical MWC
39 conditions. The fuel supply and air supply will also be
40 adjusted as needed to approach the target H₂O concentration
41 in exhaust gas in Table C.2-1.

42

C.2-8

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

10

11 C.2.4.3.4 Blanks

12

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

18

19 C.2.4.4 Process Monitoring

20

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

27

28 **Table C.2-2. Combustion Test Monitoring**

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

29

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

33

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

C.2-3

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1
2 The amount of material fed to the system will be verified
3 by weighing the pyroprobe insert cartridge before and after
4 each experiment.

5
6 Exhaust gas monitoring is described in Section C.2.4.5.

7
8 C.2.4.5 Exhaust Gas Monitoring

9
10 Combustion exhaust gas will be continuously monitored for
11 oxygen during each combustion test via in-line MSD or via
12 an oxygen monitor. CO₂ in exhaust gas will be monitored via
13 in-line GC, in-line MSD, or a continuous monitor; or
14 exhaust gas will be collected in Tedlar® bags for off-line
15 analysis of CO₂. Carbon monoxide (CO) in exhaust gas will
16 be monitored via in-line GC or a continuous monitor; or
17 exhaust gas will be collected in Tedlar® bags for off-line
18 analysis of CO. Tedlar® bag samples may be collected at
19 the exit of the bubblers described in Section C.2.4.6.

20
21 C.2.4.6 Exhaust Gas Sampling

22
23 Gas samples for off-line analysis will be collected as
24 described in Appendix D.1.

25
26 A minimum of 60 mL of bubbler aqueous solution composite is
27 expected from each combustion test. Of this, a minimum of
28 45 mL will be directed to PFOA analysis, and the remainder
29 will be directed to fluoride ion analysis.

30
31 C.2.4.7 Exhaust Gas Analysis

32
33 C.2.4.7.1 Fluoride Ion

34
35 A portion of the composite bubbler aqueous solution sample
36 from each combustion test collected as described in Section
37 C.2.4.6 will be analyzed for fluoride ion via ion
38 chromatography.

39
40 C.2.4.7.2 PFOA

41
42 A portion of the composite bubbler aqueous solution sample
43 from each combustion test collected as described in Section
44 C.2.4.6 will be analyzed for PFOA via LC/MS/MS as described
45 in Appendix D.2.

46
47 As described in Appendix D.2, sample results less than

C.2-10

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1 method detection limit (MDL) will be reported as ND, sample
2 results between MDL and the limit of quantitation (LOQ)
3 will be reported as NQ, and numerical values will not be
4 reported for such samples.

5
6 Due to background levels of PFOA, the analytical laboratory
7 will only report numerical values for PFOA concentration in
8 the aqueous solution greater than or equal to the LOQ.
9 This is required to assure that the reported concentration
10 value is attributable to the sample rather than to
11 background.

12
13 C.2.4.8 Test Substance Sampling & Analysis

14
15 See Section C.2.1. (Elemental Analysis)

16
17 C.2.5 Reporting of Results

18
19 C.2.5.1 Elemental Analysis Results

20
21 The results of elemental analysis of the test substance
22 composites (as noted in Section C.2.1) will be reported.
23 The laboratory reports will be included in an appendix to
24 the test report.

25
26 C.2.5.2 Combustion Stoichiometry Results

27
28 Combustion stoichiometry (as noted in Section C.2.2)
29 calculations will be included in an appendix to the test
30 report.

31
32 C.2.5.3 TGA Results

33
34 TGA graphical results for test substance composites (as
35 noted in Section C.2.3) will be included in an appendix to
36 the test report.

37
38 C.2.5.4 Combustion Test Results

39
40 C.2.5.4.1 Process Monitoring

41
42 Process monitoring data (as noted in Section C.2.4.4)
43 recorded for each combustion test will be reported in
44 tabular form.

45
46 C.2.5.4.2 Exhaust Gas Monitoring

47

C.2-11

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1 Exhaust gas O₂, CO and CO₂ monitoring results will be
2 reported as the integrated or average value for each
3 combustion test. CO will be reported in terms of parts per
4 million by volume (ppmv). O₂ and CO₂ will be reported in
5 terms of percent by volume (%).

6
7 C.2.5.4.3 Exhaust Gas Analytical Results

8
9 Results of analyses noted in Section C.2.4.5.2 will be
10 reported for each combustion test.

11
12 The analytical result for each analyte in Section C.2.4.5.2
13 will be reported in terms of concentration (mass per
14 volume) in the bubbler aqueous solution. For each analyte,
15 this value will be used with the associated exhaust gas
16 volume to compute an exhaust gas concentration and with the
17 associated test substance mass to compute mass of analyte
18 per mass of test substance composite.

19
20 C.2.5.4.3.1 Fluoride

21
22 Fluoride ion in the exhaust gas will be reported on the
23 basis of mass of fluoride ion per mass of test substance
24 composite. The corresponding hydrogen fluoride value for
25 each will also be computed and reported for reference.

26
27 C.2.5.4.3.2 PFOA

28
29 PFOA results for the bubbler aqueous solution samples will
30 be reported as described in Section C.2.4.7.2. PFOA
31 results for associated blanks will also be reported.

32
33 If present in the bubbler aqueous solution at a
34 concentration above the matrix-specific LOQ, PFOA in the
35 exhaust gas will be reported on the basis of mass of PFOA
36 per mass of test substance composite.

37
38 C.2.5.5 Test Substance Results

39
40 Elemental compositions will be reported as indicated in
41 Section C.2.4.6.1 above.

42
43 C.2.5.6 Release Assessment

44
45 In the event that PFOA is found in the exhaust gas bubbler
46 aqueous solution at a concentration above the LOQ for the
47 three runs for a given test substance composite, then the

1.2-12

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1 potential for release from full-scale municipal and/or
2 medical waste incineration (including application of air
3 pollution controls) of the subject material in the U.S.
4 will be assessed to inform the basis for possible next
5 steps.

6
7 This assessment will consider a number of factors such as
8

- 9 • PFOA results from this testing program reported per
10 Section C.2.5.4.3.2,
- 11 • estimated concentration of subject material in feed to
12 applicable type(s) of full-scale waste incinerators
13 (based on such information as Appendix A.2, supplemental
14 information on material applications, and available
15 information on hydrogen fluoride concentration in waste
16 incinerator exhaust as upper bound), and
- 17 • use and abatement effectiveness of common post-combustion
18 air pollution control equipment (e.g., lime scrubbing,
19 carbon adsorption) employed by typical operating full-
20 scale waste incinerators.

21
22 See Appendix E.2 for the draft outline of the Release
23 Assessment report in case this report is to be submitted.

C.2-13

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APPENDIX D

ATTACHMENTS AND REFERENCED MATERIALS

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

1 **APPENDIX D.1**
2 **EXHAUST GAS SAMPLING VIA BUBBLERS**
3

4 Gas samples for off-line analysis will be collected from a
5 vent line off the interface of the thermal reactor system
6 described in Appendix C.2.4. Flexible (silicone or
7 equivalent) tubing will connect the vent line and a set of
8 bubblers.
9

10 Gas absorption via these bubblers will provide aqueous
11 solution (of documented content) to analyze for prescribed
12 parameters. Two to four bubblers (low pressure drop
13 impingers) in series will be used. Each bubbler will
14 contain a predetermined amount of aqueous solution, and the
15 total amount of solution at the beginning of each test run
16 will be a minimum of 55 mL. The temperature of the gas
17 exiting the last bubbler will be monitored.
18

19 An additional ~~empty~~ bubbler (which is empty) will be added
20 to the front end of this series of bubblers to serve as a
21 knock-out pot if calculations or preliminary measurements
22 indicate that greater than 10 mL of water will be produced
23 during the testing for a given material.
24

25 Upon completion of sample collection, the amount in each
26 bubbler will be weighed and recorded, and the contents of
27 the bubblers will be composited for subsequent analysis.
28 Additionally, the flexible tubing will be rinsed with 5 mL
29 of HPLC water to collect potential condensate in the tubing;
30 this rinsate will be combined with the bubbler composite
31 prior to analysis.
32

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

D.1-1

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

3
4 D.2.1 Introduction

5
6 Samples to be analyzed for PFOA in this study will be
7 subjected to Liquid Chromatography with Tandem Mass
8 Spectrometry (LC/MS/MS) in accordance with "Method of
9 Analysis for the Determination of Ammonium
10 Perfluorooctanoate (APFO) in Water Revision 1" (Exygen
11 method) revised per the section-by-section comments listed
12 in Section D.2.4 below. These revisions are necessary to
13 adapt a method originally developed for liter quantity
14 water samples to samples related to testing described in
15 Appendix C.

16
17 The testing programs described in Appendix C are expected
18 to generate samples of aqueous solution, methanol (e.g., as
19 used for extraction or rinsing), and corresponding blanks.
20 The expected sample size for aqueous solution samples (from
21 exhaust gas bubbler sample collection) available for
22 analysis via this method is approximately 50 mL.

23
24 D.2.2 Method Summary

25
26 PFOA is extracted from water using a disposable C₁₈ solid
27 phase extraction (SPE) cartridge. PFOA is eluted from the
28 cartridge with methanol. Quantification of PFOA is
29 accomplished by electrospray liquid chromatography/tandem
30 mass spectrometry (LC/MS/MS) analysis.

31
32 D.2.3 Reporting

33
34 The target limit of quantitation (LOQ) for this study with
35 this method is 50 ng/L based on prior work with water
36 samples where an 8-fold concentration via extraction using
37 C₁₈ SPE cartridge has been demonstrated. The actual LOQ
38 will be matrix dependent; for samples (e.g., methanol
39 rinsate) where the 8-fold concentration cannot be
40 performed, the target LOQ for this study is 400 ng/L.

41
42 Sections 4.5.4 and 5 of the Exygen method explain reporting
43 for field samples such as bubbler aqueous solution
44 composites, which are distinct from blanks and spikes, such
45 as the types of samples generated by the testing programs
46 described in Appendix C, as follows:

47
D.2-1

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1 Field samples in which either no peaks or peaks less than the
2 MDL are detected at the corresponding analyte retention time
3 will be reported as ND (not detected). Samples in which
4 peaks are detected at the corresponding analyte retention
5 time that are less than the LOQ and greater than or equal to
6 the MDL will be reported as NQ (not quantifiable).
7
8 Therefore, sample results less than method detection limit
9 (MDL) will be reported as ND, and sample results between
10 MDL and the limit of quantitation (LOQ) will be reported as
11 NQ. Numerical values will not be reported for such
12 samples. Only concentrations above the LOQ, where the
13 reported concentration is attributable to the sample rather
14 than to background, are reported with numerical values.
15
16 Additionally, if the ~~analyte~~ PFOA anion is found in a
17 sample at a concentration above the LOQ for the matrix but
18 is less than 5 times the concentration found in the
19 associated blank, the result will be flagged and treated as
20 ND.
21
22 D.2.4 Study-Specific Comments on the Method
23

Section	Comment
1	<ul style="list-style-type: none">• The concentration of PFOA found will be reported directly and the mathematical conversion for reporting as APFO mentioned in the 4th sentence of the 2nd paragraph will not be performed.• Since the 8-fold concentration described in the 2nd sentence 4th paragraph (which forms the basis for the LOQ in the 3rd paragraph and the MDL in the 4th paragraph) is dependent on having a minimum of 40 mL of aqueous sample amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of the method, the LOQ and MDL in the method will be a factor of 8 higher than reported where less than 40 mL of sample is available or where the sample is not amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of the method (e.g., methanol).
<u>3.3 Note at top of page 8</u>	<ul style="list-style-type: none">• <u>The note stating "Equivalent materials may be substituted for those specified in this method if they can be shown to produce satisfactory results" will not be used in the analysis for this testing program.</u>

<u>3.3</u> <u>Notes,</u> <u>Note 1</u>	<ul style="list-style-type: none">• <u>The following text will be used in place of Note 1 with respect to the PFOA analysis conducted for this testing program:</u> <u>In order to avoid contamination, the use of disposable labware (tubes, pipets, etc.) is required.</u>
<u>3.3</u> <u>Notes,</u> <u>Note 4</u>	<ul style="list-style-type: none">• <u>The following text will be used in place of Note 4 with respect to the PFOA analysis conducted for this testing program:</u> <u>Solvents (e.g., methanol) used for this analysis must be checked for the presence of contaminants by LC/MS/MS before use.</u>
3.5 opening text prior to 3.5.1	<ul style="list-style-type: none">• Where the available amount of sample is expected to be much less than 1 liter, insufficient sample is available to prepare the fortified matrix spikes described in the opening text of section 3.5. In this case, the analytical standards discussed in this opening text will be limited to two purposes <u>since the third purpose (matrix spike) stated in the method cannot be done.</u>
4.3, item b	<ul style="list-style-type: none">• Where the available amount of sample is expected to be less than 80 mL (= 2 * 40), the replicate extraction noted in the first sentence of this item cannot be performed.• If the sample is not an aqueous sample amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of the method, then section 4.4 is skipped such that the sample is analyzed directly. (Note: For such samples, the LOQ and MDL will be 8 times higher than <u>the values quoted in the method.</u>)
4.3, item c	<ul style="list-style-type: none">• As noted in comment on section 3.5 opening text above, fortified matrix spikes will not be prepared when the available amount of sample is much less than 1 liter.• Where the available amount of sample is expected to be less than 80 mL (= 2 * 40), the conditional repeat fortification and extraction described in the third sentence of this item cannot be performed.
4.4	<ul style="list-style-type: none">• Extraction using the C₁₈ SPE cartridge requires a suitable aqueous sample. This extraction and the corresponding 8-fold concentration

D.2-3

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	pointed out in the NOTE at the end of this section cannot be performed on non-aqueous (e.g., methanol) samples.
4.4, item 1	<ul style="list-style-type: none">• In order to measure out the 40 mL mentioned in this item, it is necessary to have at least 45 mL of sample to enable pipet transfer.
4.5.4, item g	<ul style="list-style-type: none">• A storage stability study for PFOA in water performed independently of the development of the method indicates that PFOA may be stored in glass, polystyrene, polypropylene, or polyethylene containers without measurable degradation for up to 68 days prior to extraction. Therefore, the total holding time between sample collection and analysis for aqueous PFOA samples in this study may exceed the 14 day limit noted in the first sentence of this item provided that the sample is not held for greater than 68 days unless additional storage stability testing justifies a longer hold time.
4.6, item 3	<ul style="list-style-type: none">• As noted in comment on section 3.5 opening text above, fortified matrix spikes will not be prepared when the available amount of sample is much less than 1 liter. In this case, acceptance criteria for matrix spike recoveries will not be considered.
5, item c	<ul style="list-style-type: none">• The calculation in equation 3 in this section will not be performed since it is not necessary to convert the PFOA analytical results to APFO for this study.

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D.2.5 Reference

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

D.2-4

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76

1 **APPENDIX D.3**
2 **WICKBOLD TORCH METHOD FOR TOTAL FLUORINE**

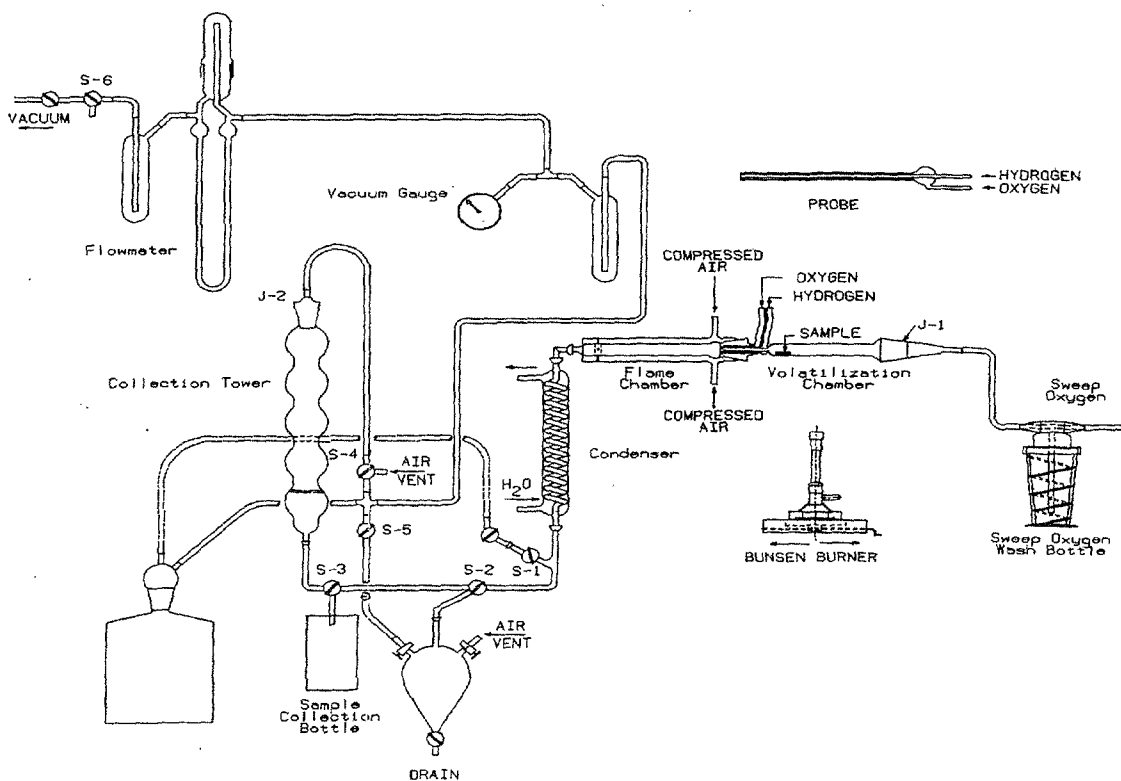
3
4 D.3.1 Introduction

5
6 "The carbon-fluorine bond is exceptionally strong, and
7 extremely vigorous conditions are needed for quantitative"
8 analysis of fluorine in organic compounds. (Kissa, 1998)
9 The "most vigorous" technique for measurement of fluorine
10 in organic compounds is "combustion in an oxyhydrogen
11 flame" referred to as the Wickbold torch. (Kissa, 1998)

12
13 D.3.2 Apparatus

14
15 A typical configuration for the Wickbold oxyhydrogen torch
16 apparatus as described by Sweetser (1956) is shown in
17 Figure D.3-1.

18
19
20 **Figure D.3-1. Wickbold Oxyhydrogen Torch Apparatus**



D.3-1

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1 D.3.3 Method Description

2

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

5

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

14

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

17

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

24

25 D.3.4 Safety Considerations

26

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

33

34 D.3.5 References

35

36 Kissa, E. "Analysis of Anionic Fluorinated Surfactants",
37 Chapter 8 in Anionic Surfactants: Analytical Chemistry -
38 2nd Edition, Revised and Expanded, edited by John Cross.
39 Marcel Dekker Surfactant Science Series, volume 73, 1998.

40

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

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APPENDIX D.4

Waste Incineration and Operation Conditions

* To be provided by the FMG.... (NOTE: Not available on 1/13/04)

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APPENDIX E

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

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APPENDIX E.1

OUTLINE FOR INTERIM REPORTING

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

OPPT Docket ID No: OPPT-2003- 0071

Date of Interim Report: [date]

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

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

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

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

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

{Note: Robert Giraud to consider the need to incorporate the following, as appropriate:

- PFOA results from this testing program reported per Section C.2.5.4.3.2,
- estimated concentration of subject material in feed to applicable type(s) of full-scale waste incinerators (based on such information as Appendix A.2, supplemental information on material applications, and available information on hydrogen fluoride concentration in waste incineration exhaust as upper bound), and
- use and abatement effectiveness of common post-combustion air pollution control equipment (e.g., lime scrubbing, carbon adsorption) employed by typical operating full-scale waste incinerators.}

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

1.0 Introduction

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

2.0 Summary of study results

- A listing of compounds collected at the targeted temperature

3.0 Discussion

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

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

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- 1 • A description of the extrapolation
- 2 • A description of any assumptions used
- 3 • Any unique qualitative or quantitative descriptors of the test, the testing
- 4 equipment, and the results deemed necessary for informative review of the test
- 5 and test results.
- 6
- 7 5.0 Sensitivity Analysis
- 8
- 9 • Assessment of the impact of variability/uncertainty (quantitative and qualitative)
- 10 in each parameter on the modeling results.
- 11 6.0 Conclusions
- 12 7.0 References

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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 ??

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

Date

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

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