TAB # 2

Appendices A - F[1]

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[1] This section of the ECA document contains the Appendices to the ECA. Additional changes and corrections to the ECA document were identified after completion of the Public Review Process. EPA set in place a process whereby the needed changes(s) were made using strike-out with hand written correction(s) on each page (i.e., replacement page) of the ECA where such changes were needed. Each signatory was asked to acknowledge their concurrence by initialing and dating every change on each ECA replacement page. To maintain continuity within the document, TAB #2 contains replacement pages initialed by EPA only. The initialed replacement pages from each Signatory Company are included under TABs #4 - #7 of this document.

APPENDIX A.1

LIST OF CHEMICAL COMPONENTS OF THE COMPOSITES[1]

The following table lists the seventeen (17) 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, contains 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.

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

FLUOROPOLYMERS SUBJECT TO THIS ECA		
No.	**CAS # / DCN # / or ACC #[2]**	**Chemical Name**
1	CAS # 9002-84-0	Ethene, tetrafluoro-, homopolymer
2	CAS # 25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene)
3	CAS # 26655-00-5	Propane,1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl0oxy]-, polymer with tetrafluoroethene
4	CAS # 25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene
5	DCN # 63040000026	ETFE
6	CAS #35560-16-8	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene
7	CAS #9011-17-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene
8	CAS #54675-89-7	1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene
9	CAS #27029-05-6	1-Propene, polymer with tetrafluoroethene
10	CAS #26425-79-6	Ethene, tetrafluoro-, polymer with trifluoro(trifluoroethoxy)ethene
11	CAS #9010-75-7	Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene
12	CAS #31784-04-0	Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene
13	DCN # 6304000018A	Polytetrafluoroethylene
14	DCN # 6304000018B	Polytetrafluoroethylene
15	DCN # 6304000018C	Fluoroelastomer
16	DCN # 6301000018D	Fluoroelastomer
17	ACC # 137678	Low Temperature Fluoroelastomer

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[2] EPA uses a variety of numerical identification systems for tracking chemicals. These include Chemical Abstract Service Registry numbers (CAS) (assigned to non-confidential listed chemicals), pre-manufacture notice (PMN) numbers (assigned by EPA when chemicals enter EPA's new chemical review process, document control numbers (DCN) (assigned by the Confidential Business Information Center for EPA tracking), and Accession (ACC) numbers (provided by EPA when a chemical identity requires protection as TSCA CBI). In addition, Polymer Exemption products will not have a TSCA Inventory ID number but may have a commercial trade identity.

APPENDIX A.2

RATIONALE FOR SELECTING COMPOSITES TO BE TESTED

Review of Figure A.2-l demonsrates that fluoropolymers industry products can be divided into 3 broad categories 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:

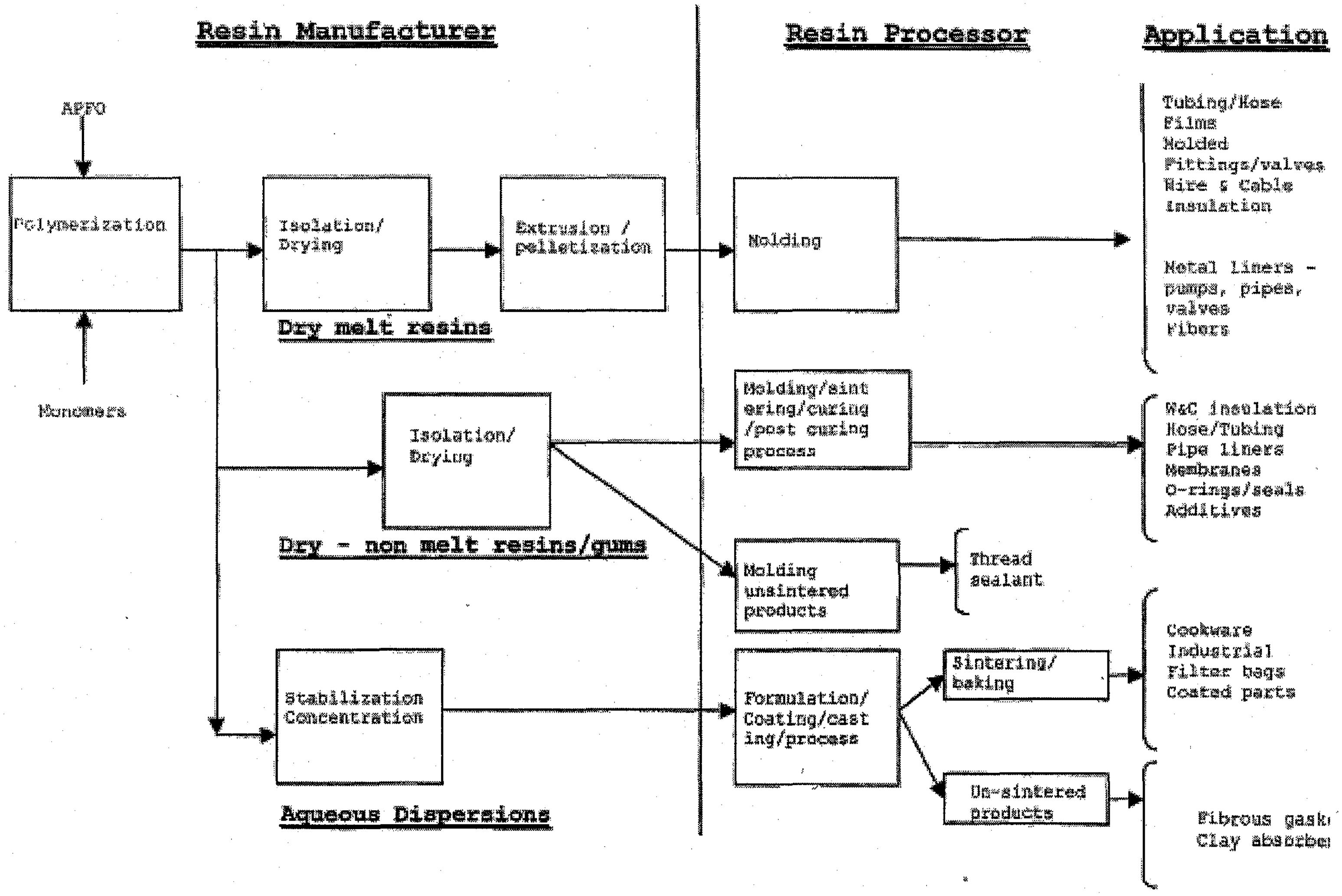
- Dry melt resins
  1. FEP, PFA, THV, ETFE, HTE

- Dry non-melt resins and gums
  2. Dry non-melt resins
  3. Fluoroelastomers (dry non-melt 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.

Figure A.2-1. Fluoropolymer Industry Overview



APPENDIX A.3
COMPOSITION OF COMPOSITES TO BE TESTED

The four composite test substances for this test program are presented below in Table A.3-I with the fluoropolymer types, CAS numbers, and associated monomers for these fluoropolymers. Each fluoropolymer used in each relevant test substance composite will have been made using APFO.

Table A.3-1. Test Substance Composites by Type

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

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

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APPENDIX A.4
PREPARATION OF COMPOSITES TO BE TESTED

4.1 Approach

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

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

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

Table A.4-1. Example for Compositing Across Companies & Types

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

4.2 Preparation

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

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

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

4.2.1 Composite 1

Dry non-melt resins are available in powder form. Equal weights of the powder form of each of the two types of components (following the approach in the example for Composite Z in Section 4.1 above) will be mixed together in dry form to yield Composite 1.

4.2.2 Composite 2

FEP, PFA ETFE awf 10/6/

~~EEP~~, ~~PEA,~~ THV, ~~ETEE,~~ and HTE dry melt resins are available in powder form. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1 above) will be mixed together in dry form to yield Composite 2.

4.2.3 Composite 3

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

a) Equal weights of each component (following the approach in example for Composite Z in Section 4.1) will be mixed on a rubber mill to produce a homogenous slab of preset thickness to yield Composite 3.

Or

b) Each component of Composite 3 will be cyrogenically cooled (to make the elastomers brittle) and size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together in dry form to yield Composite 3.

4.2.4 Composite 4

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

a) Equal weights (on a dry solids basis) of each component in aqueous dispersion form (following the approach in example for Composite Z in Section 4.1) will be mixed together in liquid form. Solids will be separated from the resulting liquid composite to yield low water content (i.e., drip free) fine solids.

Or

b) Solids will be separated from liquid for each component of Composite 4 to yield low water content (i.e., drip free) fine solids for each component. Equal weights of the solids form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together to yield Composite 4.

4.3 Verification

In order to assure that composite samples in this testing program have been made up of clearly identified materials, the preparation of the composites will include formal Chain of Custody procedures. A chain of custody form will be included with each component material going into the composite to show the identity of the component material and each transfer of custody from its point of origination to preparation of the composite.

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

For documentation, the facility preparing a given composite will generate a report to be submitted to EPA with the final report for Phase II incineration testing.

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APPENDIX B.1
GUIDELINE FOR THERMOGRAVIMETRIC ANALYSIS

As described in Appendix C.2.3, thermogravimetric analysis will be conducted as part of this testing program. An available standard method (ASTM E 1868-02) has been adapted for conducting this thermogravimetric analysis to determine the temperature range required for gasification. Therefore, ASTM E 1868-02 "Standard Test Method for Loss-On-Drying by Thermogravimetry" will be used as the guideline for conducting the analysis described in Appendix C.2.3 with the following modifications for this testing program:

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

11.6	• The test specimen heating rate will be set at 10 to 25°C/min, rather than 5°C/min noted in the first sentence of this section. Pursuant to section 11.6, the temperature program rate will be documented in the report.
11.9	• Termination criteria will follow Test Method A as outlined in section 11.10.1.
11.10.1	• The "fixed period of test time" mentioned in this section will be set at 5 min.
11.10.1.1	• Loss-on-drying values will not be recorded.
12.1	• The loss-on-drying value will not be calculated.
13.1.1	• The "identification and description of the material being tested" will be consistent with the information known to the analyst.
13.1.5	• The loss-on-drying value will not be included in the report.
14.2	• This section is not applicable because the Test Method A termination criteria will be used.

Reference

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

APENDIX C.1
PFOA TRANSPORT TESTING

C.1.1 Significance

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

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

C.1.2 Experimental Plan

C.1.2.1 Base Plan

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

A PFOA standard of known purity greater than or equal to 97% will be gasified at 150 to 300 °C (based on thermogravimetric analysis of PFOA) with transfer line and reactor temperatures 0 to 150 °C higher than the gasification temperature. With the exception of temperature as noted above, the target operating conditions for the high temperature reactor of the thermal reactor system will be consistent with the conditions presented in Table C.2-1 in Appendix C.2.4.

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

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

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

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

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

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

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

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

C.1.2.2 Contingent Testing

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

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

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

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

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

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

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

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

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

C.1.3 Reporting of Results

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

If Appendix C.2 incineration testing is performed, the detailed results of Appendix C.1 transport testing will be included in the test report for Appendix C.2 incineration testing described in Appendix C.2.5. If Appendix C.2 incineration testing is not performed, the detailed results of Appendix C.1 transport testing will be provided in a test report for Appendix C.1 transport testing.

APPENDIX C.2
INCINERATION TESTING

C.2.1 ELEMENTAL ANALYSIS

C.2.1.1 Introduction

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

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

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

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

C.2.1.2 Total Fluorine

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

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

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

C.2.1.3 Carbon and Hydrogen

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

C.2.1.3.1 Theoretical Determination

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

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

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

C.2.1.3.2 Empirical Determination

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

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

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

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

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

C.2.1.4 Moisture

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

Moisture is determined by measuring the loss of weight of the sample when heated under controlled conditions. A representative sample is weighed and placed in a crucible (or dish) and evaporated to dryness in an air or nitrogen atmosphere at a defined temperature setpoint (e.g., 103 °C to 105 °C) in the range of 100 °C to 125 °C. The moisture value is calculated as the loss in weight (difference between the starting weight of sample and the final weight of sample) divided by the starting weight of sample. Similarly, a solids value can be calculated as the final weight of sample divided by the starting weight of sample.

C.2.2 COMBUSTION STOICHIOMETRY

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

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

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

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

$$C + O_2 \rightarrow CO_2$$

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

$$S + O_2 \rightarrow SO_2$$

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

$$H_2 + Cl_2 \rightarrow 2HCl$$

$$H_2 + F_2 \rightarrow 2HF$$

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

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

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

$$N_2 \rightarrow N_2$$

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

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

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

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

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

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

C.2.3 THERMOGRAVIMETRIC ANALYSIS

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

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

C.2.4 Combustion Testing

C.2.4.1 Test Objective

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

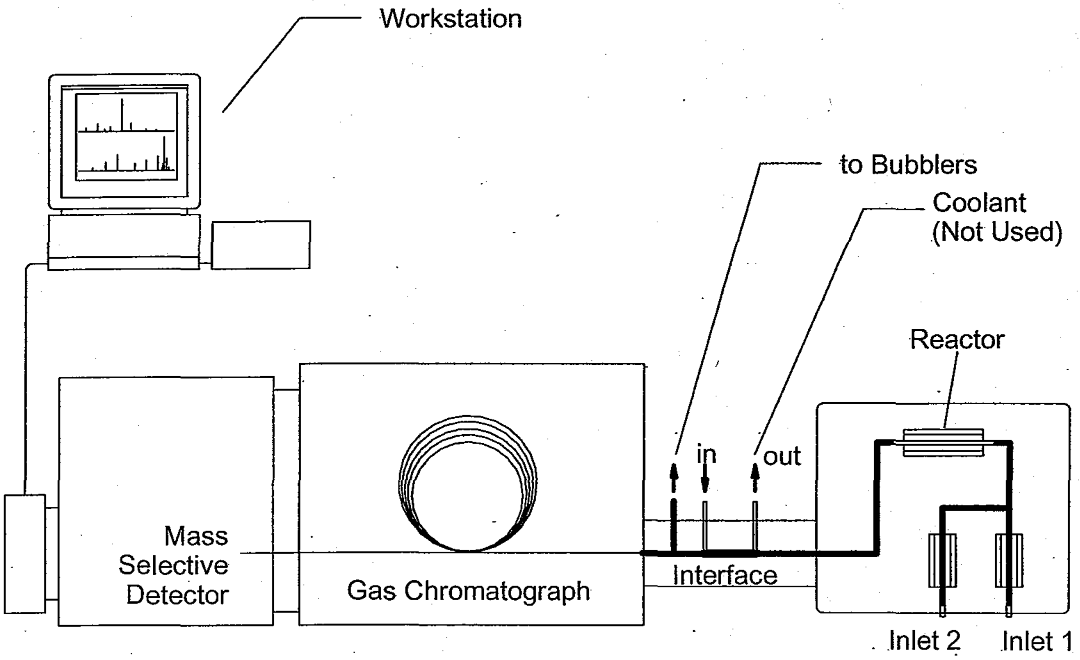
C.2.4.2 Experimental Apparatus

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

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

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

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



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

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

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

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

C.2.4.3 Combustion Test Experimental Conditions

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

C.2.4.3.1 Combustion Air

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

C.2.4.3.2 Fuel

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

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

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

C.2.4.3.3 Operating Conditions

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

TABLE C.2-1. COMBUSTION TEST TARGET OPERATING CONDITIONS

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

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

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

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

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

C.2.4.3.4 Blanks

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

C.2.4.4 Process Monitoring

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

TABLE C.2-2. COMBUSTION TEST MONITORING

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

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

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

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

Exhaust gas monitoring is described in Section C.2.4.5.

C.2.4.5 Exhaust Gas Monitoring

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

C.2.4.6 Exhaust Gas Sampling

Gas samples for off-line analysis will be collected as described in Appendix D.1, revised as necessary pursuant to Appendix C.1.2.2 if applicable.

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

C.2.4.7 Exhaust Gas Analysis

C.2.4.7.1 Fluoride Ion

A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section C.2.4.6 will be analyzed for fluoride ion via ion chromatography using EPA Method 300.0.

C.2.4.7.2 PFOA

A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section

C.2.4.6 will be analyzed for PFOA via LC/MS/MS as described in Appendix D.2.

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

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

C.2.5 Reporting of Results

C.2.5.1 Elemental Analysis Results

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

C.2.5.2 Combustion Stoichiometry Results

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

C.2.5.3 TGA Results

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

C.2.5.4 Combustion Test Results

C.2.5.4.1 Process Monitoring

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

C.2.5.4.2 Exhaust Gas Monitoring

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

C.2.5.4.3 Exhaust Gas Analytical Results

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

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

C.2.5.4.3.1 Fluoride

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

C.2.5.4.3.2 PFOA

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

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

C.2.5.5 Release Assessment

In the event that PFOA is reported for the exhaust gas bubbler aqueous solution at a concentration at or above the LOQ (as defined in Appendix D.2) for two or more of the three runs for a given test substance composite, a release assessment report for the full-scale waste incineration of products represented by the test substance composite will be prepared following the outline in Appendix E.2 and will be included in the test report.

C.2.5.6 Test Report Outline

The outline for the test report is presented in Appendix E.3. All reporting discussed in Sections C.2.5.1 through C.2.5.5 will be included in this test report, as applicable.

APPENDIX D.1
EXHAUST GAS SAMPLING VIA BUBBLERS

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

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

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

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

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

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

D.2.1 Introduction

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

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

D.2.2 Method Summary

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

D.2.3 Reporting

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

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

> Field samples in which either no peaks or peaks

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

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

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

D.2.4 Study-Specific Comments on the Method

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

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

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

D.2.5 Reference

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

APPENDIX D.3
WICKBOLD TORCH METHOD FOR TOTAL FLUORINE

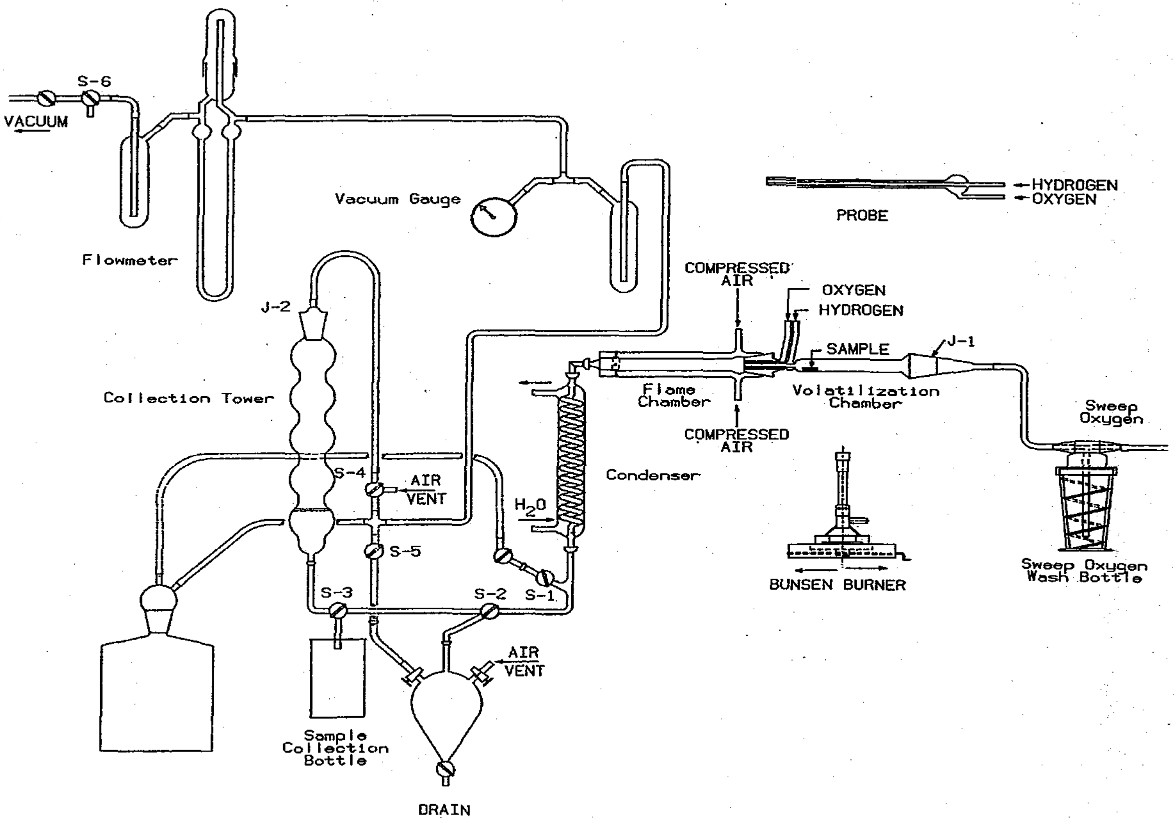
D.3.1 Introduction

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

D.3.2 Apparatus

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

FIGURE D.3-1. WICKBOLD OXYHYDROGEN TORCH APPARATUS



D.3.3 Method Description

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

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

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

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

D.3.4 Safety Considerations

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

D.3.5 References

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

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

APENDIX D.4
WASTE INCINERATION AND OPERATION CONDITIONS

Polymers of the sort being investigated in this testing program may be present at trace to low concentrations in the feedstreams to municipal waste combustors and/or medical waste incinerators in the U.S.

D.4.1 Types of Incinerators

D.4.2.1 Municipal Waste Combustors

According to the Integrated Waste Services Association (IWSA), there are a total of 98 waste-to-energy facilities operating municipal waste combustors (MWCs) in the U.S. as of 2002. (IWSA 2002) Table D.4-1 summarizes the number and annual capacity of these units by type of technology employed.

Table D.4-1. MWCs in 2002

Type	Number of Facilities	Annual Capacity (million Ton/year)	Fraction of Waste
Mass Burn	68	22.5	76.5%
Refused Derived Fuel (RDF)	18	6.4	21.8%
Modular	12	0.5	1.7%
Total	98	29.4	100.0%

D.4.1.2 Hospital/Medical/Infectious Waste Incinerators

Although earlier reports indicated approximately 2400 medical waste incinerators in the U.S. in the 1990s burning approximately 846 thousand tons of hospital and medical/infectious waste (EPA 1997), the current EPA Office of Air Quality, Planning, and Standards (OAQPS) inventory indicates that there are 116 hospital/medical/infectious waste incinerators (HMIWIs) in the U.S. as of July 28, 2003. (EPA 2003)

This represents a greater than 90% reduction in the number of operating HMIWIs in the U.S. Many medical waste incinerators were closed rather than upgraded to meet new emission standards, as hospitals improved their programs to segregate infectious ("red bag") waste burned in HMIWIs from non-infectious ("black bag") waste handled as municipal solid waste after it leaves the hospital. Consequently, the amount of segregated infectious waste burned in HMIWIs is expected to be less than 0.3 million tons per year.

EPA notes that over 97% of medical waste incinerators are controlled air modular units (EPA 2000a). Recent communication with EPA OAQPS indicates that virtually all existing HMIWIs are controlled air modular (two-chamber) units.

D.4.2 Incinerator Operating Conditions

Many incinerators for municipal solid waste are designed to operate in the combustion zone at 1800 °F (982 °C) to 2000 °F (1093 °C) to ensure good combustion. (EPA 1995) EPA's new source performance standards (NSPS) and emission guidelines for both municipal waste combustors (MWCs) and hospital/medical/infectious waste incinerators (HMIWIs) are based on the use of "good combustion practices" (GCP). (EPA 1997, EPA 2000b, EPA 2000c, Van Remmen 1998)

Referring to MWCs, Donnelly notes, "Design of modern efficient combustors is such that there is adequate turbulence in the flue gas to ensure good mixing, a high-temperature zone (greater than 1000 °C) to complete burnout, and long enough residence time at high temperature (1-2 sec) for complete burnout." (Donnelly 2000) The term "flue gas" here refers to the gas above the grate.

With respect to HMIWIs, Van Remmen states "any unit which presently [prior to compliance date] has a [secondary chamber] residence time less than two seconds at 1000 °C does not meet the requirement for good combustion under the new regulations." (Van Remmen 1998)

Similarly, most MWCs operate with a 2 second gas residence time in the high temperature zone in order to assure compliance with emission standards on carbon monoxide (CO) and dioxins.

D.4.2.1 MWC Operating Conditions

D.4.2.1.1 Mass Burn MWC

Review of the IWSA Directory (IWSA 2002) indicates that almost all of these mass burn units are mass burn water wall furnaces. Nearly all mass burn water wall furnaces have reciprocating grates or roller grates to move the waste through the combustion chamber. (EPA 1996a)

Studies on the Millbury, Massachusetts mass burn water wall MWC produced gas temperature versus residence time results. (Scavuzzo, Strempek, and Strach 1990) Calculations based on Figure 6 of this paper indicate a time-averaged temperature of 2238 °F (1226 °C) over a 2 second. The corresponding gas temeperature at the 2 second level from this figure is 1750 °F (954 °C).

A report on the Warren County, New Jersey mass burn water wall MWC indicates that the design gas temperature between the grate and secondary air inject was greater than 2000 °F (1093 °C) over a gas residence time of an additional 2.2 seconds. (Schuetzenduebel and Nobles 1990) This report also shows that this MWC was designed for 2 seconds residence time above 1800 °F (982 °C) between the introduction of secondary air and the exit of the furnace section. (Schuetzenduebel and Nobles 1990) The temperature profile (Figure 21) in the temperature correlation test report (Schutzenduebel 1989) for this MWC shows the full load gas temperature at the secondary air injection point is 2650 °F, and the gas temperature at the 2-second point is 1850 °F. Therefore, testing indicates an average temperature of 2250 °F (1232 °C) over this 2 second gas residence time for the Warren County unit. A related report for the Warren County MWC by the design firm indicates that the exhaust gas oxygen concentration is nominally 10% (dry basis). (Blount Energy Resource Corp. 1989)

Information from these 2 MWCs demonstrates that the average gas temperature across a 2 second residence time for mass burn MWCs is conservatively expected to be greater than 1100 °C.

Test report data from a typical mass burn MWC (Fairfax, Virginia) indicates typical average furnace exit gas concentrations are 10.8% oxygen (dry basis) and 18.4% moisture (water). (Clean Air Engineering, 1997)

As indicated in Table D.4.1, mass burn units account for over 76% of the municipal solid waste incinerated in the U.S.

D.4.2.1.2 RDF MWC

Furnace temperatures as well as flue gas oxygen and moisture (H_2O) levels for the Mid-Connecticut RDF combustor during performance tests while operating under good combustion conditions across a range of steam loads (Finklestein and Klicius 1994) are summarized in Table D.4-2.

Table D.4-2. RDF MWC - Mid-Connecticut

Steam load	low	low	inter-mediate	inter-mediate	normal	normal	normal	high
test number	PT-13	PT-14	PT-10	PT-02	PT-09	PT-08	PT-11	PT-12
Furnace temperature (°C)	965	1004	1012	1022	1033	1015	1026	1049
flue gas O_2 (%)	10.1	9.6	9.2	9.1	7.6	7.5	7.9	6.4
flue gas moisture	12.4	11.1	12.3	15.4	15.1	16.3	14.1	16.2

The average operating conditions for this RDF unit across the range of steam loads are 1016 °C, 8.4% O_2 (dry basis), and 14.1% moisture.

Examination of the report and MWC temperature monitoring practices indicates that these temperatures are effectively combustion zone exit temperatures. Therefore, in order to determine the average MWC combustion zone temperature across a 2 second gas residence time, it is necessary to understand the time-temperature profile of the MWC.

Since waste combustion in this and most other RDF units in the U.S. involves burning on the grate (EPA 1996a) similar to the operation of mass burn MWCs, the time-temperature profile in an RDF unit is expected to be similar to that described in Section D.4.2.1.1 above. Based on this similarity and the temperatures in Table D.4-2, the average gas temperature across a 2 second residence time for RDF units is conservatively expected to be greater than 1100 °C.

As indicated in Table D.4.1, RDF units account for approximately 22% of the municipal solid waste incinerated in the U.S.

D.4.2.1.1 Modular MWC

Modular MWCs are generally small dual-chamber units, accounting for less than a total of 2% of the municipal solid waste incinerated in the U.S. in 2002. Modular MWCs are generally equipped with auxiliary fuel burners in the secondary chamber. (EPA 1996a) EPA notes that the secondary chamber exit temperature of modular MWCs is maintained at typically 980 to 1200 °C. (EPA 1996a)

A typical modular MWC in Polk County, Minnesota is operated with a gas residence time of 2 seconds, in the secondary chamber, a secondary chamber exit temperature in the range of 1800 °F (982 °C) to 2000 °F (1093 °C), flue gas oxygen concentrations in the range of 10% to 13% (dry basis), and flue gas moisture in the range of 10% to 15% (Pace Analytical 2003).

Since the secondary chamber exit temperature is expected to be the minimum gas-phase temperature for the chamber, the secondary chamber average gas temperatures for modular MWCs are expected to be 1000 °C or greater.

As indicated in section D.4.1, such modular units are generally small MWCs and account for less than a total of 2% of the municipal solid waste incinerated in the U.S.

D.4.2.1.4 MWC Summary

Considering the relative quantities of municipal waste burned annually in each type of MWC and the data in this section, typical operating conditions for the high temperature zone of most MWCs are >1000 °C average temperature across 2 second residence time with exit gas concentrations of 10% O_2 (dry basis) and >15% moisture.

D.4.2.2 HMIWI Operating Conditions

The range of temperatures for the secondary chamber of controlled air medical waste incinerators has been reported as 980 to 1200 °C. (Theodore 1990) EPA notes that auxiliary fuel (e.g., natural gas) is burned in the secondary chamber of medical waste incinerators to sustain temperatures in the range of 985 to 1095 °C and that combustion air at 150 to 250 % of the stoichiometric requirement is usually added to the secondary chamber. (EPA 2000a, EPA 1994a)

In its model plant description background document, EPA notes that the average moisture content in HMIWI flue gas was about 10 % based on available data, and EPA states "limited data show that older [HMIWI] units typically have residence times that range from essentially 0 seconds up to about 1 second." (EPA 1994b) However, as noted above, a more recent report indicates that HMIWIs still in operation have secondary chamber temperatures greater than or equal to 1000 °C with a gas residence time of 2 seconds. (Van Remmen 1998) For example, EPA studied the incinerator at Weeks Hospital in New Hampshire as a typical HMIWI with a design residence time of 2 seconds in the secondary chamber. (EPA 1996b) During this testing, the average exit secondary chamber exit temperature was 1024 °C, and the flue gas oxygen concentration was 13.5%. (dry basis)(EPA 1996b)

Review of test reports for all HMIWIs in the EPA docket for the HMIWI NSPS and EG rulemakings that are listed in EPA's current HMIWI inventory (EPA 2003) does not refute Van Remmen's statement above on residence time and temperature and indicates HMIWI flue gas oxygen concentrations for these units in the range of 10 to 15% (dry basis)and stack moisture concentrations as high as 30% (after wet scrubbing). (Environmental Laboratories Inc. 1993, EPA 1996, HDR Engineering 1994a, HDR Engineering 1994b, METCO Environmental 1992, Technical Services, Inc. 1993, Technical Services, Inc. 1994a, Technical Services, Inc. 1994b) Apparently, the older HMIWIs referred to in EPA's model plant description background document either have been shut down or upgraded to operate with secondary chamber exit temperatures higher than 1000 °C at a gas residence time of 2 seconds.

Secondary chamber temperature of HMIWIs is monitored near the secondary chamber outlet. (EPA 1994) Hence, when the auxiliary burner (located on the end opposite from the outlet) is in use, the average gas temperature in an HMIWI secondary chamber is greater than the outlet temperatures noted above. Therefore, secondary chamber average gas temperatures for HMIWIs are expected to be 1000 °C or greater with a gas residence time of 2 seconds.

In summary, typical operating conditions for the secondary chamber of operating HMIWIs in the U.S. are 1000 °C average temperature across 2 second residence time with exit gas concentrations of 13% O_2 (dry basis) and >10% moisture.

D.4.3 Pollution Control Equipment

Over 99% of large MWC capacity operates with a spray dryer absorber/scrubber. (IWSA 2003) Approximately 80% of large MWC capacity operates using carbon injection as part of the pollution control system. (IWSA 2003) Due to requirements in the NSPS (EPA 2000b) and EG (EPA 200c) for small MWCs, small MWCs planning continued operation are generally upgrading or have upgraded their pollution control equipment to add spray dryer absorbers or other acid gas control and carbon injection.

Review of EPA's HMIWI inventory (EPA 2003) indicates that essentially all HMIWIs have some form of wet or dry scrubbing for acid gas control.

D.4.4 Summary

Approximately 30 million tons per year of municipal solid waste was combusted in the United States annually in waste-to-energy municipal waste combustors in 2003. Approximately 0.3 million tons per year of segregated medical waste was combusted annually in the United States in hospital/medical/infectious waste incinerators in 2003. Considering the relative amounts of waste combusted annually, typical operating conditions for waste incineration in the U.S. across these two classes of units are as follows:

Average Temperature	>1000 °C
Residence Time	$\geq$2 sec
O_2 concentration in exhaust gas	10% (dry basis)
H_2O concentration in exhaust gas	15%

EPA emission regulations currently in place or in place by 2005 require that operating municipal waste combustors and hospital/medical/infectious waste incinerators have or will have air pollution control equipment such as wet or dry scrubbing for acid gas control.

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APPENDIX E.1 (Fluoropolymers)
OUTLINE FOR INTERIM PROGRESS REPORTING

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

OPPT Docket ID No: OPPT-2003-0071

Date of Interim Report: [date]

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

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

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

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

4) Other information relevant to the progress of the testing program: (If none indicate N/A)

APPENDIX E.2
OUTLINE FOR RELEASE ASSESSMENT REPORT

As described in Appendix C.2.5.5 of this ECA, if PFOA is reported for the exhaust gas bubbler aqueous solution at a concentration at or above the LOQ (as defined in Appendix D.2) for two or more of the three runs for a given test substance composite, then the potential for release from full-scale municipal and/or medical waste incineration, as applicable, (including application of air pollution controls) of products represented by the test substance composite in the United States will be assessed to put the data into perspective. At a minimum, the report will follow the general outline described below and will state assumptions, document the basis for the assumptions made, quantitatively estimate the variability of calculated estimates (based on the variability of the parameters in the evaluation), and qualitatively discuss the uncertainty of calculated estimates.

1.0 Introduction

- Statement of objective for combustion testing of test substance composites.

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

2.0 Summary of study results

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

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

3.0 Discussion

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

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

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

- The relevance of the subject test substance composite to MWCs and/or medical waste incinerators.

- The estimated concentration of the subject test substance composite to the applicable type(s) of waste incinerator. Available information on hydrogen fluoride concentration in waste incinerator exhaust can provide the basis for an upper bound on this estimated concentration.

- A description of the extrapolation.

- A description of any assumptions used.

- Any unique qualitative or quantitative descriptors of the test, the testing equipment, and the results deemed necessary for informative review of the test and test results.

5.0 Sensitivity Analysis

- Assessment of the impact of variability (quantitative) and uncertainty (qualitative) in each parameter on the evaluation results.

6.0 Conclusions

7.0 References

APPENDIX E.3
OUTLINE OF TEST REPORT*,**

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Executive Summary

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    5.1 PFOA
    5.2 Total Fluorine
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        5.3.2 Exhaust Gas Monitoring
        5.3.2 Exhaust Gas Sampling and Analysis
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            5.3.2.2 Fluoride
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III. Appendices

- Quality Assurance Report(s)

- Report(s) from Compositing Facility(ies) (provided Phase II is performed)

- Reports from Analytical Laboratories

- Release Assessment per Appendix E.2, if applicable

* Test Report will include this information (as applicable) but not necessarily in this order.

** References to literature in this report will include full citations.

APPENDIX F

UNITED STATES
ENVIRONMENTAL PROTECTION AGENCY

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

EPA Docket No. OPPT - 2003 - 0071

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

Date

Susan B. Hazen
Principal Deputy Assistant Administrator
Office of Prevention, Pesticides and Toxic Substances

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TAB # 3

Company Specific Signature Pages Containing Confidential Business Information[1]

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[1] The Company specific signature pages contained in this section of the ECA are not publically available because one or more of the Signatory Companies have asserted that information contained on these pages is considered by them to be entitled for protection as TSCA confidential business information (CBI) (see Part XIV. D. of the ECA regarding confidentiality of information). The sanitized version of the signature pages are available in the Public Version of this document under TAB #3 (see Part XXIV of the ECA).

TAB # 4

Replacement Pages for AGC Chemicals Americas, Inc.[2]

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[2] Additional changes and corrections to the ECA document were identified after completion of the Public Review Process. EPA set in place a process whereby the needed changes(s) were made using strike-out with hand written correction(s) on each page (i.e., replacement page) of the ECA where such changes were needed. Each signatory was asked to acknowledge their concurrence by initialing and dating every change on each ECA replacement page. The ECA document contained under TAB # 1 contains the replacement pages originated by EPA. The pages under this Tab contain those replacement pages co-initialed by EPA and AGC Chemicals Americas, Inc.

(D) Aqueous Fluoropolymer Dispersions Composite #4: (containing: ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy) ethene, CAS No. 31784-04-0;~~ Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3- [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0;~~ 1-Propene, 1,1,2,3,3,3- hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; and polytetrafluoroethylene, DCN No. 63040000018~~C~~B).[8]

MR 1/25/05 JW 1/4/0
MR 1/25/05 JW 1/4/0
MR 1/25/05 JW 1/4/0

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

III. OBLIGATION OF SIGNATORY COMPANIES

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

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

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[8] EPA uses a variety of numerical identification systems for tracking chemicals. These include Chemical Abstract Service Registry numbers (CAS) (assigned to non-confidential listed chemicals), pre-manufacture notice (PMN) numbers (assigned by EPA when chemicals enter EPA's new chemical review process, document control numbers (DCN) (assigned by the Confidential Business Information Center for EPA tracking), and Accession (ACC) numbers (provided by EPA when a chemical identity requires protection as TSCA CBI.). In addition, Polymer Exemption products will not have a TSCA Inventory ID number but may have a commercial trade identity.

to seek judicial review of any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA.

XXII. RESERVATION OF RIGHTS BY COMPANIES

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

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

XXIII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR

The Principal Test Sponsor is:

Fluoropolymer Manufacturers Group
Allen Weidman
The Society of the Plastics Industry, Inc.
1801 K Streert, N.W., Suite 600K
Washington, DC 20006
202-974-5233

The Companies subject to this ECA are:

AGC Chemicals Americas, Inc.
229 East 22nd Street,
Bayonne, NJ 07002

Daikin America, Inc.
20 Olympic Drive,
Orangeburg, NY 10962

Dyneon, LLC
6744 33rd Street,
Oakdale, MN 55128

E.I. du Pont de Nemours and Company
~~Route 141 and Henry Clay~~ 1007 MARKET STRE
Wilmington, DE ~~19880-0711~~ 19898

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Copy #2 - Public Version **Contains No Confidential Business Information**

XXIV. SIGNATURE

TEST SPONSOR

Dyneon, LLC [1, 2]

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

Name:	George H. Millet
Title:	Director, Quality, Environment, Health and Safety
Address:	6744 33rd Street, Oakdale, MN 55128
Phone Number:	651-733-5637

ECA Subject Chemicals for Dyneon, LLC *

Entry	Composite	CAS Registry #	CAS 9CI Name
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry non-melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
5	Dry melt Fluoropolymer Resin	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene
6	Dry melt Fluoropolymer Resin		

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

[2] Dyneon, LLC is obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

XXIV. SIGNATURE TEST SPONSOR

E.I. du Pont de Nemours and Company[1, 2]

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

Name: David W. Boothe
Title: Strategic Planning Manager - DuPont Fluoro~~solutions~~ Products
Address: ~~Route 141 & Henry Clay,~~ Chestnut Run 702 4417 Lancaster Pike Wilmington, DE ~~19880-0711~~ 19805
Phone Number: 302-999-4091

ECA Subject Chemicals for E. I. du Pont de Nemours and Company

Entry	Composite	CAS Registry #	CAS 9CI Name
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #31784-04-0	Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene
5	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
6	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
7	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene

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[1] Data in the table lists the chemical(s) and composite contributions for which E.I. du Pont de Nemours and Company is responsible. The Company developed these data in response to EPA's letter of January 6, 2004.

[2] E.I. du Pont de Nemours and Company is not obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

Continued: ECA Subject Chemicals for Dyneon, LLC

Entry	Composite	CAS Registry #	CAS 9CI Name
7	Dry melt Fluoropolymer Resin	CAS #35560-16-8	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene
8	Dry non-melt Fluoroelastomer Gum	CAS #27029-05-6	1-Propene, polymer with tetrafluoroethene
9	Dry non-melt Fluoroelastomer Gum	CAS #54675-89-7	1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene
10	Dry non-melt Fluoroelastomer Gum	CAS #26425-79-6	Ethene, tetrafluoro-, polymer with trifluoro(trifluoromethoxy) ethene
11	Dry non-melt Fluoroelastomer Gum	CAS #9010-75-7	Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene
12	Dry non-melt Fluoroelastomer Gum	[redacted]	[redacted]
13	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
14	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
15	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
16	Aqueous Dispersion	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene

* Entries "X'd" out indicate redacted information claimed as CBI by the Company.

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APPENDIX A.3
COMPOSITION OF COMPOSITES TO BE TESTED

The four composite test substances for this test program are presented below in Table A.3-1 with the fluoropolymer types, CAS numbers, and associated monomers for these fluoropolymers. Each fluoropolymer used in each relevant test substance composite will have been made using APFO.

Table A.3-1. Test Substance Composites by Type

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

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

4.2.2 Composite 2

~~EEP,~~ FEP, ~~PEA,~~ PFA, THV, ~~ETEE,~~ ETFE and HTE dry melt resins are available in powder form. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1 above) will be mixed together in dry form to yield Composite 2.

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4.2.3 Composite 3

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

a) Equal weights of each component (following the approach in example for Composite Z in Section 4.1) will be mixed on a rubber mill to produce a homogenous slab of preset thickness to yield Composite 3.

Or

b) Each component of Composite 3 will be cyrogenically cooled (to make the elastomers brittle) and size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together in dry form to yield Composite 3.

4.2.4 Composite 4

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

a) Equal weights (on a dry solids basis) of each component in aqueous dispersion form (following the approach in example for Composite Z in Section 4.1) will be mixed together in liquid form. Solids will be separated from the resulting liquid composite to yield low water content (i.e., drip free) fine solids.

Or

b) Solids will be separated from liquid for each component of Composite 4 to yield low water content (i.e., drip free) fine solids for each component. Equal weights of the solids form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together to yield Composite 4.

TAB # 5

Replacement Pages for Daikin America, Inc.[1]

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[1] Additional changes and corrections to the ECA document were identified after completion of the Public Review Process. EPA set in place a process whereby the needed changes(s) were made using strike-out with hand written correction(s) on each page (i.e., replacement page) of the ECA where such changes were needed. Each signatory was asked to acknowledge their concurrence by initialing and dating every change on each ECA replacement page. The ECA document contained under TAB # 1 contains the replacement pages originated by EPA. The pages under this Tab contain those replacement pages co-initialed by EPA and Daikin America, Inc.

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(D) Aqueous Fluoropolymer Dispersions Composite #4: (containing: ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)-ethene, CAS No. 31784-04-0;~~ Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3- [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0;~~ 1-Propene, 1,1,2,3,3,3- hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; and polytetrafluoroethylene, DCN No. 63040000018~~C~~B).[8]

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

III. OBLIGATION OF SIGNATORY COMPANIES

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

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

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[8] EPA uses a variety of numerical identification systems for tracking chemicals. These include Chemical Abstract Service Registry numbers (CAS) (assigned to non-confidential listed chemicals), pre-manufacture notice (PMN) numbers (assigned by EPA when chemicals enter EPA's new chemical review process, document control numbers (DCN) (assigned by the Confidential Business Information Center for EPA tracking), and Accession (ACC) numbers (provided by EPA when a chemical identity requires protection as TSCA CBI). In addition, Polymer Exemption products will not have a TSCA Inventory ID number but may have a commercial trade identity.

to seek judicial review of any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA.

XXII. RESERVATION OF RIGHTS BY COMPANIES

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

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

XXIII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR

The Principal Test Sponsor is:

Fluoropolymer Manufacturers Group
Allen Weidman
The Society of the Plastics Industry, Inc.
1801 K Streert, N.W., Suite 600K
Washington, DC 20006
202-974-5233

The Companies subject to this ECA are:

AGC Chemicals Americas, Inc.
229 East 22nd Street,
Bayonne, NJ 07002

Dyneon, LLC
6744 33rd Street,
Oakdale, MN 55128

Daikin America, Inc.
20 Olympic Drive,
Orangeburg, NY 10962

E.I. du Pont de Nemours and Company
~~Route 141 and Henry Clay~~ 1007 MARKET STREE
Wilmington, ~~DE 19880-0711~~ 19898

mp 10/6/04
SD 1/19/05

XXIV. SIGNATURE

TEST SPONSOR

Dyneon, LLC [1, 2]

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

Name: George H. Millet
Title: Director, Quality, Environment, Health and Safety
Address: 6744 33rd Street, Oakdale, MN 55128
Phone Number: 651-733-5637

ECA Subject Chemicals for Dyneon, LLC *

Entry	Composite	CAS Registry #	CAS 9CI Name
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry non-melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
5	Dry melt Fluoropolymer Resin	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene
6	Dry melt Fluoropolymer Resin		

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SD 1/19/05

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

[2] Dyneon, LLC is obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

Continued: ECA Subject Chemicals for Dyneon, LLC

Entry	Composite	CAS Registry #	CAS 9CI Name
7	Dry melt Fluoropolymer Resin	CAS #35560-16-8	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene
8	Dry non-melt Fluoroelastomer Gum	CAS #27029-05-6	1-Propene, polymer with tetrafluoroethene
9	Dry non-melt Fluoroelastomer Gum	CAS #54675-89-7	1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene
10	Dry non-melt Fluoroelastomer Gum	CAS #26425-79-6	Ethene, tetrafluoro-, polymer with trifluoro(trifluoromethoxy) ethene
11	Dry non-melt Fluoroelastomer Gum	CAS #9010-75-7	Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene
12	Dry non-melt Fluoroelastomer Gum		
13	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
14	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
15	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
16	Aqueous Dispersion	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene

* Entries "X'd" out indicate redacted information claimed as CBI by the Company.

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wP 10/6/01 -SD 1/19/05

XXIV. SIGNATURE **TEST SPONSOR**

E.I. du Pont de Nemours and Company [1], [2]

WP 10/6/05;
SD 7/19/05

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

Name: David W. Boothe
Title: Strategic Planning Manager - DuPont Fluoro~~solutions~~ Products
Address: ~~Route 141 & Henry Clay,~~ 4417 Lancaster Pike Chestnut Run 702 Wilmington, DE ~~19880-0711~~ 19805
Phone Number: 302-999-4091

ECA Subject Chemicals for E. I. du Pont de Nemours and Company

Entry	Composite	CAS Registry #	CAS 9CI Name
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #31784-04-0	Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene
5	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
6	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
7	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene

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[1] Data in the table lists the chemical(s) and composite contributions for which E.I. du Pont de Nemours and Company is responsible. The Company developed these data in response to EPA's letter of January 6, 2004.

[2] E.I. du Pont de Nemours and Company is not obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

APPENDIX A.3
COMPOSITION OF COMPOSITES TO BE TESTED

The four composite test substances for this test program are presented below in Table A.3-1 with the fluoropolymer types, CAS numbers, and associated monomers for these fluoropolymers. Each fluoropolymer used in each relevant test substance composite will have been made using APFO.

Table A.3-1. Test Substance Composites by Type

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

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-SD 1/19/05

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

4.2.2 Composite 2

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FEP, PFA ETFE

~~EEP,~~ ~~PEA,~~ THV, ~~ETEE,~~ and HTE dry melt resins are available in powder form. Equal weights SD :/19/05 of the powder form of each component (following the approach in the example for Composite Z in Section 4.1 above) will be mixed together in dry form to yield Composite 2.

4.2.3 Composite 3

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

a) Equal weights of each component (following the approach in example for Composite Z in Section 4.1) will be mixed on a rubber mill to produce a homogenous slab of preset thickness to yield Composite 3.

Or

b) Each component of Composite 3 will be cyrogenically cooled (to make the elastomers brittle) and size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together in dry form to yield Composite 3.

4.2.4 Composite 4

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

a) Equal weights (on a dry solids basis) of each component in aqueous dispersion form (following the approach in example for Composite Z in Section 4.1) will be mixed together in liquid form. Solids will be separated from the resulting liquid composite to yield low water content (i.e., drip free) fine solids.

Or

b) Solids will be separated from liquid for each component of Composite 4 to yield low water content (i.e., drip free) fine solids for each component. Equal weights of the solids form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together to yield Composite 4.

TAB # 6

Replacement Pages for Dyneon, LLC[1]

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[1] Additional changes and corrections to the ECA document were identified after completion of the Public Review Process. EPA set in place a process whereby the needed changes(s) were made using strike-out with hand written correction(s) on each page (i.e., replacement page) of the ECA where such changes were needed. Each signatory was asked to acknowledge their concurrence by initialing and dating every change on each ECA replacement page. The ECA document contained under TAB # 1 contains the replacement pages originated by EPA. The pages under this Tab contain those replacement pages co-initialed by EPA and Dyneon LLC.

(D) Aqueous Fluoropolymer Dispersions Composite #4: (containing: ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0;~~ Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3- [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0;~~ 1-Propene, 1,1,2,3,3,3- hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; and polytetrafluoroethylene, DCN No. 630400000018~~C~~B).[8]

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William Myers 1/13/c

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William Myers 1/13/0

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

III. OBLIGATION OF SIGNATORY COMPANIES

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

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

---

[8] EPA uses a variety of numerical identification systems for tracking chemicals. These include Chemical Abstract Service Registry numbers (CAS) (assigned to non-confidential listed chemicals), pre-manufacture notice (PMN) numbers (assigned by EPA when chemicals enter EPA's new chemical review process, document control numbers (DCN) (assigned by the Confidential Business Information Center for EPA tracking), and Accession (ACC) numbers (provided by EPA when a chemical identity requires protection as TSCA CBI). In addition, Polymer Exemption products will not have a TSCA Inventory ID number but may have a commercial trade identity.

to seek judicial review of any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA.

XXII. RESERVATION OF RIGHTS BY COMPANIES

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

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

XXIII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR

The Principal Test Sponsor is:

Fluoropolymer Manufacturers Group
Allen Weidman
The Society of the Plastics Industry, Inc.
1801 K Streert, N.W., Suite 600K
Washington, DC 20006
202-974-5233

The Companies subject to this ECA are:

AGC Chemicals Americas, Inc.
229 East 22nd Street,
Bayonne, NJ 07002

Daikin America, Inc.
20 Olympic Drive,
Orangeburg, NY 10962

Dyneon, LLC
6744 33rd Street,
Oakdale, MN 55128

E.I. du Pont de Nemours and Company
~~Route 141 and Henry Clay~~ 1007 MARKET STRE
Wilmington, DE ~~19880-0711~~ 19898

wp 10/6/04
William Myers 10/11/04

XXIV. SIGNATURE

TEST SPONSOR

Dyneon, LLC [1, 2]

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

Name:	George H. Millet
Title:	Director, Quality, Environment, Health and Safety
Address:	6744 33rd Street, Oakdale, MN 55128
Phone Number:	651-733-5637

ECA Subject Chemicals for Dyneon, LLC *

Entry	Composite	CAS Registry #	CAS 9CI Name
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry non-melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
5	Dry melt Fluoropolymer Resin	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene
6	Dry melt Fluoropolymer Resin		

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10/11/04

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

[2] Dyneon, LLC is obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

Continued: ECA Subject Chemicals for Dyneon, LLC

Entry	Composite	CAS Registry #	CAS 9CI Name
7	Dry melt Fluoropolymer Resin	CAS #35560-16-8	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene
8	Dry non-melt Fluoroelastomer Gum	CAS #27029-05-6	1-Propene, polymer with tetrafluoroethene
9	Dry non-melt Fluoroelastomer Gum	CAS #54675-89-7	1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene
10	Dry non-melt Fluoroelastomer Gum	CAS #26425-79-6	Ethene, tetrafluoro-, polymer with trifluoro(trifluoromethoxy) ethene
11	Dry non-melt Fluoroelastomer Gum	CAS #9010-75-7	Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene
12	Dry non-melt Fluoroelastomer Gum	[redacted]	[redacted]
13	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
14	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
15	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
16	Aqueous Dispersion	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene

* Entries "X'd" out indicate redacted information claimed as CBI by the Company.

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Copy #2 - Public Version **Contains No Confidential Business Information**

XXIV. SIGNATURE **TEST SPONSOR**

E.I. du Pont de Nemours and Company[1, 2]

Company technical contact person for handling correspondence marked as "Confidential" WP 10/6/04 William Myers 10/6/04

Name: David W. Boothe
Title: Strategic Planning Manager - DuPont Fluoro~~solutions~~ Products
Address: Chestnut Run 702 4417 Lancaster Pike ~~Route 141 & Henry Clay,~~ Wilmington, DE ~~19880-0711~~ 19805
Phone Number: 302-999-4091

ECA Subject Chemicals for E. I. du Pont de Nemours and Company			
Entry	**Composite**	**CAS Registry #**	**CAS 9CI Name**
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #31784-04-0	Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene
5	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
6	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
7	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene

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[1] Data in the table lists the chemical(s) and composite contributions for which E.I. du Pont de Nemours and Company is responsible. The Company developed these data in response to EPA's letter of January 6, 2004.

[2] E.I. du Pont de Nemours and Company is not obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

APPENDIX A.3
COMPOSITION OF COMPOSITES TO BE TESTED

The four composite test substances for this test program are presented below in Table A.3-1 with the fluoropolymer types, CAS numbers, and associated monomers for these fluoropolymers. Each fluoropolymer used in each relevant test substance composite will have been made using APFO.

Table A.3-1. Test Substance Composites by Type

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

wmf 10/6/04

William Myers
10/11/04

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

William Myers 10/1/0
awf 10/6/0

4.2.2 Composite 2

FEP, PFA ETFE
~~EEP,~~ ~~PFA,~~ THV, ~~ETFE,~~ and HTE dry melt resins are available in powder form. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1 above) will be mixed together in dry form to yield Composite 2.

4.2.3 Composite 3

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

a) Equal weights of each component (following the approach in example for Composite Z in Section 4.1) will be mixed on a rubber mill to produce a homogenous slab of preset thickness to yield Composite 3.

Or

b) Each component of Composite 3 will be cyrogenically cooled (to make the elastomers brittle) and size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together in dry form to yield Composite 3.

4.2.4 Composite 4

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

a) Equal weights (on a dry solids basis) of each component in aqueous dispersion form (following the approach in example for Composite Z in Section 4.1) will be mixed together in liquid form. Solids will be separated from the resulting liquid composite to yield low water content (i.e., drip free) fine solids.

Or

b) Solids will be separated from liquid for each component of Composite 4 to yield low water content (i.e., drip free) fine solids for each component. Equal weights of the solids form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together to yield Composite 4.

TAB # 7

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

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[1] Additional changes and corrections to the ECA document were identified after completion of the Public Review Process. EPA set in place a process whereby the needed changes(s) were made using strike-out with hand written correction(s) on each page (i.e., replacement page) of the ECA where such changes were needed. Each signatory was asked to acknowledge their concurrence by initialing and dating every change on each ECA replacement page. The ECA document contained under TAB # 1 contains the replacement pages originated by EPA. The pages under this Tab contain those replacement pages co-initialed by EPA and E.I du Pont de Nemours and Company.

(D) Aqueous Fluoropolymer Dispersions Composite #4: (containing: ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)-ethene, CAS No. 31784-04-0;~~ Ethene, tetrafluoro-, homopolymer, CAS No. 9002-84-0; 1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene), CAS No. 25067-11-2; Propane, 1,1,1,2,2,3,3-heptafluoro-3- [(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene, CAS No. 26655-00-5; ~~Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene, CAS No. 31784-04-0;~~ 1-Propene, 1,1,2,3,3,3- hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene, CAS No. 25190-89-0; and polytetrafluoroethylene, DCN No. 630400000018~~C~~B).[8]

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

III. OBLIGATION OF SIGNATORY COMPANIES

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

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

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[8] EPA uses a variety of numerical identification systems for tracking chemicals. These include Chemical Abstract Service Registry numbers (CAS) (assigned to non-confidential listed chemicals), pre-manufacture notice (PMN) numbers (assigned by EPA when chemicals enter EPA's new chemical review process, document control numbers (DCN) (assigned by the Confidential Business Information Center for EPA tracking), and Accession (ACC) numbers (provided by EPA when a chemical identity requires protection as TSCA CBI). In addition, Polymer Exemption products will not have a TSCA Inventory ID number but may have a commercial trade identity.

to seek judicial review of any rule that may be adopted by EPA that imposes requirements to test any of the fluoropolymer chemicals listed in Appendix A.1 to this ECA.

XXII. RESERVATION OF RIGHTS BY COMPANIES

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

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

XXIII. IDENTITY OF THE COMPANIES AND PRINCIPAL TEST SPONSOR

The Principal Test Sponsor is:

Fluoropolymer Manufacturers Group
Allen Weidman
The Society of the Plastics Industry, Inc.
1801 K Streert, N.W., Suite 600K
Washington, DC 20006
202-974-5233

The Companies subject to this ECA are:

AGC Chemicals Americas, Inc.
229 East 22nd Street,
Bayonne, NJ 07002

Daikin America, Inc.
20 Olympic Drive,
Orangeburg, NY 10962

Dyneon, LLC
6744 33rd Street,
Oakdale, MN 55128

E.I. du Pont de Nemours and Company
~~Route 141 and Henry Clay~~ 1007 MARKET STREI
Wilmington, DE ~~19880-0711~~ 19898

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DS 10/19/04

XXIV. SIGNATURE

TEST SPONSOR

Dyneon, LLC [1, 2]

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

Name: George H. Millet
Title: Director, Quality, Environment, Health and Safety
Address: 6744 33rd Street, Oakdale, MN 55128
Phone Number: 651-733-5637

ECA Subject Chemicals for Dyneon, LLC *

Entry	Composite	CAS Registry #	CAS 9CI Name
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry non-melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
5	Dry melt Fluoropolymer Resin	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene
6	Dry melt Fluoropolymer Resin		

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

[2] Dyneon, LLC is obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

Continued: ECA Subject Chemicals for Dyneon, LLC

Entry	Composite	CAS Registry #	CAS 9CI Name
7	Dry melt Fluoropolymer Resin	CAS #35560-16-8	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with ethene and tetrafluoroethene
8	Dry non-melt Fluoroelastomer Gum	CAS #27029-05-6	1-Propene, polymer with tetrafluoroethene
9	Dry non-melt Fluoroelastomer Gum	CAS #54675-89-7	1-Propene, polymer with 1,1-difluoroethene and tetrafluoroethene
10	Dry non-melt Fluoroelastomer Gum	CAS #26425-79-6	Ethene, tetrafluoro-, polymer with trifluoro(trifluoromethoxy) ethene
11	Dry non-melt Fluoroelastomer Gum	CAS #9010-75-7	Ethene, chlorotrifluoro-, polymer with 1,1-difluoroethene
12	Dry non-melt Fluoroelastomer Gum	[redacted]	[redacted]
13	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
14	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
15	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-[(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
16	Aqueous Dispersion	CAS #25190-89-0	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with 1,1-difluoroethene and tetrafluoroethene

* Entries "X'd" out indicate redacted information claimed as CBI by the Company.

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XXIV. SIGNATURE

TEST SPONSOR

E.I. du Pont de Nemours and Company [1], [2]

Company technical contact person for handling correspondence marked as "Confidential" WP 10/6/6

JCS 1/12/05

Name: David W. Boothe
Title: Strategic Planning Manager - DuPont Fluoro~~solutions~~ Products
Address: ~~Route 141 & Henry Clay,~~ 4417 Lancaster Pike Chestnut Run 702 Wilmington, DE ~~19880-0711~~ 19805
Phone Number: 302-999-4091

ECA Subject Chemicals for E. I. du Pont de Nemours and Company			
Entry	**Composite**	**CAS Registry #**	**CAS 9CI Name**
1	Dry non-melt Fluoropolymer Resin	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
2	Dry melt Fluoropolymer Resin	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
3	Dry melt Fluoropolymer Resin	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene
4	Dry melt Fluoropolymer Resin	CAS #31784-04-0	Ethene, tetrafluoro-, polymer with trifluoro(pentafluoroethoxy)ethene
5	Aqueous Dispersion	CAS #9002-84-0	Ethene, tetrafluoro-, homopolymer
6	Aqueous Dispersion	CAS #25067-11-2	1-Propene, 1,1,2,3,3,3-hexafluoro-, polymer with tetrafluoroethene
7	Aqueous Dispersion	CAS #26655-00-5	Propane, 1,1,1,2,2,3,3-heptafluoro-3-(trifluoroethenyl)oxy]-, polymer with tetrafluoroethene

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[1] Data in the table lists the chemical(s) and composite contributions for which E.I. du Pont de Nemours and Company is responsible. The Company developed these data in response to EPA's letter of January 6, 2004.

[2] E.I. du Pont de Nemours and Company is not obligated under this ECA to perform Phase I PFOA Transport Testing (see Part III. C. and VII.A. of this ECA).

APPENDIX A.3
COMPOSITION OF COMPOSITES TO BE TESTED

The four composite test substances for this test program are presented below in Table A.3-1 with the fluoropolymer types, CAS numbers, and associated monomers for these fluoropolymers. Each fluoropolymer used in each relevant test substance composite will have been made using APFO.

Table A.3-1. Test Substance Composites by Type

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

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

4.2.2 Composite 2

FEP, PFA ETFE 10/6/0 10/19/04

~~EEP,~~ ~~PEA,~~ THV, ~~ETEE,~~ and HTE dry melt resins are available in powder form. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1 above) will be mixed together in dry form to yield Composite 2.

4.2.3 Composite 3

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

a) Equal weights of each component (following the approach in example for Composite Z in Section 4.1) will be mixed on a rubber mill to produce a homogenous slab of preset thickness to yield Composite 3.

Or

b) Each component of Composite 3 will be cyrogenically cooled (to make the elastomers brittle) and size-reduced (e.g., ground) to produce powder. Equal weights of the powder form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together in dry form to yield Composite 3.

4.2.4 Composite 4

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

a) Equal weights (on a dry solids basis) of each component in aqueous dispersion form (following the approach in example for Composite Z in Section 4.1) will be mixed together in liquid form. Solids will be separated from the resulting liquid composite to yield low water content (i.e., drip free) fine solids.

Or

b) Solids will be separated from liquid for each component of Composite 4 to yield low water content (i.e., drip free) fine solids for each component. Equal weights of the solids form of each component (following the approach in the example for Composite Z in Section 4.1) will be mixed together to yield Composite 4.