0PPJ-2003-007/-0092



Robert J Giraud <Robert.J.Giraud@US A.dupont.com>

03/02/04 12:40 PM

To: Rich Leukroth/DC/USEPA/US@EPA cc: david.menotti@shawpittman.com Subject: Re: Appendix F cover page

Rich,

Thank you for resending this as a WPD file.

In the interest of time, I have gone ahead and placed the requested language (with minor edits noted below) into the pdf file for Appendices B through G as new page F-0 as requested this morning. I have not been able to speak with David since receiving this text. I do not recall having seen this text before receiving it from you after this morning's call. I would like your assurance that you will review it with David between the time he is back in the office this afternoon (approximately 2:30 pm March 2 per his secretary) and when the documents get sent out for distribution. (I will be tied up on a different call this afternoon.)

The minor edits are as follows: changed font size from 11 to 10 to enable this preamble language to fit on 1 page, revised "Quality Assurance" in the title to "QAPP" to better clarify that the issue is about the needed content of the QAPP rather than with ongoing plans for quality assurance, and dropped the words "the need for and/or" in the last sentence because David has made it very clear that Appendix F is needed. I am counting on you to touch base with David as noted above to see if he has other requested revisions to page F-0 (and to related text for the transmittal letter) prior to distribution.

(See attached file: Appendices B through G draft 2-27-04-revised-2.pdf)

I have also made the other revisions discussed during this morning's drafting committee conference call. And, as we agreed on the phone this set of appendices as well as the previously transmitted Appendix A pdf files are staying with 2-27-04 as the date even though the ECA cover document may be dated 3-2-04.

If you have any questions or comments, please let me know.

Best Regards,

Robert Giraud

P.S. I am looking forward to hearing from you regarding our meeting with Barbara L. on Wed. March 10 or Friday March 5.

Leukroth.Rich@epamail.epa.gov on 03/02/2004 10:26:56 AM

To: Robert J Giraud/AE/DuPont@DuPont cc: david.menotti@shawpittman.com Subject: Appendix F cover page

As discussed during this mornings call.... here is the page that we agreed to insert before Appendix F.

(See attached file: 2views on QAPP.pdf)

In thinking further.... It would probably be a good idea to have the most current date on the headers for these documents going to the Interested Parties. If this is a simple task for you please make the changes to read 3-2-04. I'll follow up at my end. Thanks.

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(See attached file: 2views on QAPP.pdf)

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Appendices B through G draft 2-27-04-revised-2 2views on QAPP.pdf

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1 APPENDIX B.1

2 GUIDELINE FOR THERMOGRAVIMETRIC ANALYSIS

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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:

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Section	Modification
2.1	• Standard practices at the University of Dayton Research Institute (UDRI) may be used as references throughout the standard in place of the ASTM standards noted in this section.
4.1	• The loss-on-drying value specified in the second through fifth sentences of this section will not be recorded.
7.1.3	 The programming rate of the furnace will be set at 10 to 25°C/min, rather than 5°C/min. Pursuant to section 11.6, the temperature program rate will be documented in the report. The isothermal temperature within the range of 25 to
	1000°C will be maintained ±3°C, rather than ±2°C.
7.1.4	• The specimen atmosphere control system will be capable of supplying dry air in addition to "inert dry gas (usually purified grade nitrogen)".
7.1.7	• The temperature program rate will be set at 10 to 25°C/min, rather than 5°C/min. Pursuant to section 11.6, the temperature program rate will be documented in the report.
	 The temperature program rate will be controlled to within the range of ±3°C/min, rather than ±0.1°C/min.
	 Within the range of 25 to 1000°C, the isothermal temperature will be maintained within ±3°C, rather than ±2°C.
11.4	 The mass of the test specimen noted in the first sentence of this section will be 0.005 to 5 mg, rather than 10±1 mg (i.e., 9 to 11 mg).
11.6	• The test specimen heating rate will be set at 10 to 25°C/min, rather than 5°C/min noted in the first sentence of this section. Pursuant to section 11.6, the temperature program rate will be documented in the report.
11.9	• Termination criteria will follow Test Method A as outlined in section 11.10.1.

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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.

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11 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 <u>service@astm.org</u>. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

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1 APENDIX C.1 2 PFOA TRANSPORT TESTING 3 4 C.1.1 Significance 5 6 Testing will be performed to verify that potential PFOA 7 emissions from the combustion testing described in Appendix 8 C.2 can be quantitatively transported from the high 9 temperature reactor into the exhaust gas sampling apparatus 10 (aqueous solution bubblers). 11 12 Acceptable PFOA transport will be demonstrated if the 13 transport efficiency (as computed in one or more of the 14 formulas below) is greater than or equal to 70%. 15 16 C.1.2 Experimental Plan 17 18 C.1.2.1 Base Plan 19 Transport of PFOA across the laboratory-scale thermal 20 reactor system described in Appendix C.2.4 and into the 21 exhaust gas bubblers described in Appendix D.1 will be 22 23 quantitatively determined as an indication of transport 24 from the high temperature reactor into the bubblers. 25 A PFOA standard of known purity greater than or equal to 26 97% will be gasified at 150 to 250 °C (based on 27 thermogravimetric analysis of PFOA) with transfer line and 28 reactor temperatures 0 to 100 °C higher than the 29 gasification temperature. 30 31 32 Three replicate transport efficiency test runs will be conducted. A minimum of one blank run will be conducted 33 34 prior to each transport efficiency test run. 35 36 The sample size of the PFOA standard to be gasified will be less than 5 mg. The reactor exhaust gas will be collected 37 38 into bubbler aqueous solution as described in Appendix D.1 (including an HPLC water rinse of the flexible tubing [used 39 40 to connect the thermal reactor system and the bubbler assembly] into the aqueous solution composite), which will 41 42 be analyzed for PFOA as described in Appendix D.2. Tn order to provide a second way of demonstrating quantitative 43 transport, this aqueous solution composite will also be 44 analyzed for total fluorine as described in Appendix D.3. 45 46 (Testing for total fluorine is included due to possibility 47 of thermal degradation of PFOA under transport test

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conditions.) Therefore, for this transport testing the 1 amount of PFOA fed to the thermal reactor system will be 2 sufficiently high to assure that the total fluorine input 3 to the thermal reactor system will be greater than 140% of 4 5 the mass corresponding to the limit of quantitation (LOQ) for total fluorine in the aqueous solution composite. (The 6 7 LOQ for total fluorine in aqueous solution is much higher than the LOQ for PFOA in aqueous solution.) 8 9 The amount of PFOA and total fluorine in the thermal 10 reactor system exhaust gas will be determined via analysis 11 12 of the aqueous solution composite as noted above. 13 14 The amount of PFOA fed to the thermal reactor system will be known based on measurement prior to gasification and 15 16 will be verified by weighing the pyroprobe insert cartridge 17 before and after each test run. The amount of fluorine input to the system will be calculated from the amount of 18 PFOA fed, the known purity of the PFOA, and the known 19 fluorine fraction of the PFOA standard. 20 21 22 PFOA transport efficiency (TE) as a percentage will be 23 computed as follows: 24 25 % PFOA TE = mass of PFOA in aqueous solution composite * 100 (1)26 mass of PFOA fed to thermal reactor system 27 28 Total fluorine (TF) transport efficiency as a percentage 29 will be computed as follows: 30 Total F TE = mass of total F in aqueous solution composite * 100 (2) 31 mass of total F fed to thermal reactor system 32 33 34 C.1.2.2 Contingent Testing 35 If the transport efficiencies for both PFOA (equation 1) 36 and total fluorine (equation 2) are less than or equal to 37 70%, then additional work will be performed. 38 This additional work will be performed in a step-wise fashion to 39 determine if consideration of one or more of the following 40 procedural revisions enables achievement of 70% transport 41 efficiency as follows: 42 43 Step 1. The flexible tubing between the thermal reactor 44 45 system and the bubbler assembly from the experiment described in Section C.1.2.1 would be 46 quantitatively rinsed with methanol. This methanol 47 rinsate would be analyzed for PFOA (as described in 48

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1 Appendix D.2) and/or for total fluorine (as 2 described in Appendix D.3). Revised transport 3 efficiency (TE) as a percentage for PFOA (equation 3) and/or total fluorine (equation 4) would be 4 5 computed by including the mass of analyte in the 6 methanol rinse in the numerator as follows: 7 8 mass_{PFOA} out 9 % PFOA TE = ---- * 100 (3)10 mass_{PFOA} in 11 12 where $mass_{PFOA}$ out = mass of PFOA in bubbler 13 aqueous solution composite 14 + mass of PFOA in methanol 15 rinse 16 17 and massproa in = mass of PFOA fed to thermal 18 reactor system 19 20 masstotal F out 21 % Total F TE = ----- * 100 (4)2Ż $mass_{total F}$ in 23 24 where $mass_{total F}$ out = mass of total F in 25 bubbler aqueous 26 solution composite 27 + mass of total F in 28 methanol rinse 29 30 calculated mass of and mass_{total F} in = 31 total F in PFOA fed to 32 thermal reactor system 33 Step 2 (if necessary) The experiment described in Section 34 C.1.2.1 would be repeated with 35 , reagent(s) (e.g. NaOH) added to the 36 bubbler aqueous solution to determine 37 if reagent addition enhances analyte 38 absorption, thereby improving transport 39 efficiency. Transport efficiency would 40 be calculated using equation (1) and/or 41 (2) above. The impact of reagent 42 addition on LOQ for PFOA analysis 43 described in Appendix D.2 would be 44 45 determined. 46 47 C.1.3 Reporting of Results 48 Following completion of PFOA transport testing as described 49 in this appendix and prior to beginning incineration 50 testing described in Appendix C.2, a letter report will be 51

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1 submitted to EPA with the transport efficiency result(s) 2 and indication of what contingent testing, if any, was 3 performed.

5 If Appendix C.2 incineration testing is performed, the 6 detailed results of Appendix C.1 transport testing will be 7 included in the test report for Appendix C.2 incineration 8 testing described in Appendix C.2.5. If Appendix C.2 9 incineration testing is not performed, the detailed results 10 of Appendix C.1 transport testing will be provided in a 11 test report for Appendix C.1 transport testing.

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1 APPENDIX C.2 2 INCINERATION TESTING 3 4 C.2.1 ELEMENTAL ANALYSIS 5 6 C.2.1.1 Introduction 7 Elemental analysis as described in Section C.2.1 will be 8 performed for each test substance composite to aid in 9 preparation for combustion testing described in Section 10 11 C.2.4. 12 13 As Kissa (1998) points out, technique strongly affects analytical results for fluorinated organic compounds such 14 15 as fluorinated surfactants and fluorinated polymers due to the carbon-fluorine bond: 16 17 18 Fluorine in organic compounds is usually determined by converting organic fluorine to an 19 20 · inorganic fluoride. Various combustion methods 21 are routinely used for this purpose. However, 22 the carbon-fluorine bond is exceptionally strong, and extremely vigorous conditions are needed for 23 a quantitative mineralization. Conventional 24 25 combustion conditions used for the determination 26 of carbon and hydrogen in nonfluorinated organic 27 compounds are not adequate for a quantitative analysis of fluorinated surfactants. 28 29 Therefore, total fluorine analysis will be performed using 30 "extremely vigorous conditions" as described in Section 31 C.2.1.2, and the commercially available conventional 32 technique used for empirical determination of carbon and 33 hydrogen content (described in Section C.2.1.3) will 34 35 provide estimated values. 36 37 C.2.1.2 Total Fluorine 38 39 Each test substance composite will be characterized via analysis of total fluorine content. 40 41 42 Based on manufacturing process knowledge, the levels of total fluorine in the components of test substance 43 44 composites are orders of magnitude higher than the potential trace level of inorganic fluoride in these 45 materials. Therefore, for this test program, the total 46 organic fluorine value for each test substance composite 47

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will be considered to be the same as the total fluorine 1 2 value. 3 4 Total fluorine content will be measured via the Wickbold 5 Torch method; see Appendix D.3. 6 7 C.2.1.3 Carbon and Hydrogen 8 9 In order to provide information for stoichiometric calculations in Section C.2.2, the carbon and hydrogen 10 content of each test substance composite is needed. Based 11 12 on manufacturing process knowledge of the polymers in this program, levels of sulfur, and nitrogen are expected to be 13 less than 0.1% and to thereby have negligible effect on 14 15 stoichiometric calculations. 16 17 C.2.1.3.1 Theoretical Determination 18 19 Where the elemental composition of a test substance composite is known from the identity of the components in a 20 21 given composite, the carbon and hydrogen content of the 22 test substance composite can be calculated. 23 24 For example, where each of the components of a test 25 substance composite are polytetrafluoroethylene (PTFE), the 26 carbon and hydrogen can be determined knowing the molecular 27 formula for PTFE is $(C_2F_4)_n$ as follows: 28

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

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30 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.

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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

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1 substance composites via commercially available conventional techniques is expected to overestimate the 2 3 hydrogen content of the test substance composites. 4 5 The carbon content of the test substance composite can be measured by determining the carbon dioxide (CO₂) generated 6 7 by the oxidation of the sample. This oxidation may be accomplished by high temperature combustion, catalytic 8 combustion, or wet chemical oxidation. The CO_2 is measured 9 directly by an infrared detector or a thermal conductivity 10 11 detector, via absorption into a suitable solution (e.g., potassium hydroxide) and gravimetric determination, or by 12 13 conversion to methane for measurement via a flame 14 ionization detector. 15 16 The hydrogen content of the sample can be determined by 17 difference with knowledge of the fluorine content and carbon content of the sample where the moisture content and 18 chlorine content of the sample are negligible or known. 19 20 Alternatively, the hydrogen content of the sample is 21 measured by determining the water generated by high 22 temperature combustion of the sample. Measurement of water in the combustion gas for this analysis may be accomplished 23 24 by techniques such as use of an infrared detector or 25 absorption on a dessicant with gravimetric determination. With empirical hydrogen determination, it is important to 26 correct for the water in the combustion gas attributable to 27 the moisture content in the sample to obtain the hydrogen 28 content of the sample; see Section C.2.1.4. 29 30 Manufacturing process knowledge of the polymers will be 31 used to review the elemental analysis results and to form 32 33 the basis for interpreting non-detects. For example, if the hydrogen analytical result for a perfluorinated polymer 34 35 is less than a quantitation limit of 0.1%, then the analytical result will be replaced with 0. 36 37 38 C.2.1.4 Moisture 39 Where preparation (as described in Appendix A.4) for a 40 given test substance composite has involved dewatering, the 41 moisture (or solids) content of each such test substance 42 composite will be determined in order to provide a dry 43 basis for calculations as needed. 44 45 Moisture is determined by measuring the loss of weight of 46 the sample when heated under controlled conditions. A 47

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representative sample is weighed and placed in a crucible 1 (or dish) and evaporated to dryness in an air or nitrogen 2 atmosphere at a defined temperature setpoint (e.g., 103 °C 3 to 105 °C) in the range of 100 °C to 125 °C. The moisture 4 value is calculated as the loss in weight (difference 5 between the starting weight of sample and the final weight 6 of sample) divided by the starting weight of sample. 7 Similarly, a solids value can be calculated as the final 8 9 weight of sample divided by the starting weight of sample. 10 11 C.2.2 COMBUSTION STOICHIOMETRY 12 13 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

17 First, the weight percent values from Section C.2.1 are 18 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 (CO₂) C + O₂ \rightarrow CO₂ .

b) All sulfur (S) in feed converts to sulfur dioxide (SO₂) S + O₂ \rightarrow SO₂

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 $2H_2 + O_2 \rightarrow 2H_2O$

40 41 e) Nitrogen (N) from feed or air is emitted as molecular 42 nitrogen 43 $N_2 \rightarrow N_2$

45 Third, with these rules, the balanced chemical reaction for 46 combustion of a compound can be written.

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For example, the resulting reaction equation for a 1 hydrocarbon like methane (CH₄) is 2 3 $CH_4 + 2 O_2 \rightarrow CO_2 + 2H_2O$ 4 5 Note that the term feed in the preceding rules (a through e) includes both material being combusted and the fuel 6 7 source of hydrogen such as methane or methanol. Additionally, stoichiometric calculations as described 8 9 above presume that the compounds undergoing combustion are essentially free of inorganic constituents. 10 11 These calculations provide the theoretical amount of oxygen 12 13 needed for the overall combustion reaction for the feed based on the available information used in the 14 15 calculations. The initial estimate for the amount of oxygen to be used in combustion testing will be determined 16 from this theoretical amount with adjustments for target 17 oxygen level in thermal reactor system exhaust gas. The 18 actual amount of oxygen to be used in combustion testing 19 will be based oxygen monitoring described in Section C.2.4. 20 21 These stoichiometric calculations will also be used as > 22 needed to initially estimate and adjust experimental 23 24 conditions for combustion testing in Section C.2.4. 25 C.2.3 THERMOGRAVIMETRIC ANALYSIS 26 27 28 Thermogravimetric analysis (TGA) will be conducted to 29 determine the temperature range required for gasification of each test substance composite. TGA will be conducted in 30 flowing air from room temperature to 1000°C as described in 31 32 Appendix B.1. 33 The TGA weight-loss profile for each test substance composite 34 will be evaluated to determine the temperature at which the 35 weight loss reaches a final asymptote across the temperature 36 range investigated. This temperature corresponds to the 37 point at which no further gasification (under test 38 conditions) occurs for the material and will be considered 39 the temperature for complete gasification of the material. 40 41 42 C.2.4 Combustion Testing 43 44 C.2.4.1 Test Objective 45 46 The objective of the testing program described in Appendix C.2 is to assess the potential for waste incineration of 47

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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

9 Combustion testing will make use of the Advanced Thermal 10 Reactor System (ATRS) at the University of Dayton Research 11 Institute (UDRI). The ATRS is a laboratory-scale, non-12 flame, batch-charged, continuous flow thermal reactor 13 system. The use of this non-flame thermal reactor system 14 gives a conservative representation of full-scale waste 15 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.

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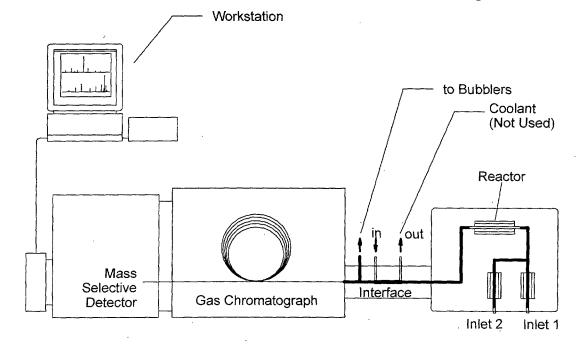
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27 Figure C.2-1. Schematic of ATRS for this Test Program



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1 The ATRS consists of a reactor assembly and in-line gas chromatograph/detector system connected via an interface. 2 The reactor assembly consists of a thermally insulated 3 4 enclosure housing the sample introduction, reactor, and 5 transfer line systems. 6 7 Sample introduction for solid materials (Inlet 1) employs a pyroprobe, a device designed to gasify samples by heating 8 them at a fixed rate. The main gas flow will also be fed 9 10 via Inlet 1, and Inlet 2 will be used to feed supplemental 11 flow. 12 During combustion tests, the transfer line between the 13 14 pyroprobe and the reactor is heated and maintained above 200 °C. The reactor is housed within its own small tube 15 furnace and may be independently heated to as high as 1100 16 (Actual conditions for this test program are presented °C. 17 in Section C.2.4.3.) The transfer line from the reactor to 18 the interface is heat traced to greater than 200 °C to 19 prevent cool regions where reactor products could otherwise 20 be lost through condensation. 21 22 The interface routes the combustion exhaust gas to the in-23 line gas chromatograph (GC) and mass selective detector 2.4 (MSD) or to sample collection for off-line analysis. For 25 combustion testing in this test program, the interface will 26 also be maintained above 200 °C. Exhaust gas monitoring for 27 this program is described in Section C.2.4.5. 28 29 C.2.4.3 Combustion Test Experimental Conditions 30 31 32 Each test substance composite will be subjected to laboratory-scale incineration using the experimental 33 apparatus described in Section C.2.4.2. 34 35 . C.2.4.3.1 Combustion Air 36 37 Synthetic air (mixture of 21% oxygen and 79% nitrogen) will 38 be used in place of compressed air to prevent potential 39 interference in the experimental system due to background 40 levels of CO_2 in compressed air. 41 42 43 C.2.4.3.2 Fuel 44 Methanol will be used, as needed, as a supplemental fuel to 45 ensure the presence of sufficient hydrogen to convert 46

fluorine to hydrogen fluoride (HF) and chlorine to hydrogen 1 chloride (HCl). 2 3 4 As noted in Municipal Solid Waste in the United States: 5 2000 Facts and Figures (EPA, 2002), paper and paper products (made from wood) make up the largest component of 6 7 municipal solid waste (MSW). The sum of paper and paper 8 products with wood in MSW makes up over 30% of MSW. 9 During the 19th century, methanol was produced from wood and 10 was known as wood alcohol. Therefore, methanol can be used 11 12 in this experimental program as a surrogate for the paper and wood fraction of MSW. 13 14 C.2.4.3.3 Operating Conditions 15 16 The target operating conditions for the high temperature 17 18 reactor during the combustion tests for each test substance 19 composite identified in Appendix A.3 are presented in Table

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22 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

23

34

24 These conditions are conservatively representative of 25 typical furnace operating conditions of municipal waste 26 combustors (MWCs) and of typical secondary chamber 27 operating temperatures for medical waste incinerators in 28 the U.S. See Appendix D.4 for supporting information. 29

30 Temperature and residence time values in Table C.2-2 will 31 be fixed setpoints for these experiments. The temperature 32 of the high temperature reactor will be controlled within 33 +10 °C to assure isothermal operation.

The amount of each test substance composite fed to the ATRS 35 in this testing program will be a measured amount less than 36 5 mg. The actual amount fed, gasification rate (determined 37 from TGA), air supply, and fuel supply will be adjusted to 38 assure that the oxygen level in the exhaust will be greater 39 than or equal to the concentration in Table C.2-1 40 throughout each test to be representative of typical MWC 41 conditions. The fuel supply and air supply will also be 42

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1 adjusted as needed to approach the target H_2O concentration 2 in exhaust gas in Table C.2-1. 3 4 The pyroprobe section final temperature (at end of temperature ramp-up) will be 750 °C or as needed to assure 5 this section is 50 to 100 °C above the highest temperature 6 7 for complete gasification across the test substance composites as determined from the TGA results; see Section 8 9 C.2.3. This is necessary to assure complete gasification of the sample of test substance composite and a common set 10 11 of experimental conditions across the test materials during 12 combustion testing. 13 C.2.4.3.4 Blanks 14 1.5 16 A minimum of one thermal blank will be run prior to each 17 set of three combustion test runs for a given test substance composite. Each thermal blank run will be at the 18 19 corresponding combustion test conditions with all feeds 20 except for the test substance. 21 22 C.2.4.4 Process Monitoring 23 24 ATRS process parameters in Table C.2-2 will be monitored for each combustion test at key points during the test as 25 noted in the table. Each combustion test will be a minimum 26 27 of 5 minutes in duration. If the duration of a combustion 28 test is greater than 15 minutes, each parameter in Table 29 C.2-2 will be recorded at least once every 15 minutes. 30 TABLE C.2-2. COMBUSTION TEST MONITORING . 31 Key Time for Recording Parameter

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

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33 Temperature-Inlet 1 will be recorded at the end of the

34 temperature ramp-up for gasification to monitor the

35 pyroprobe final temperature.

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The flow rate of the exhaust gas routed to the bubblers 1 (see Section C.2.4.5.2) will be determined based on the 2 3 flow measurements listed in Table C.2-2. 4 5 The amount of material fed to the system will be verified by weighing the pyroprobe insert cartridge before and after б 7 each experiment. 8 9 Exhaust gas monitoring is described in Section C.2.4.5. 10 11 C.2.4.5 Exhaust Gas Monitoring 12 13 Combustion exhaust gas will be continuously monitored for 14 oxygen during each combustion test via in-line MSD or via an oxygen monitor. CO_2 in exhaust gas will be monitored via 15 16 in-line GC, in-line MSD, or a continuous monitor; or exhaust gas will be collected in Tedlar® bags for off-line 17 analysis of CO₂. Carbon monoxide (CO) in exhaust gas will 18 19 be monitored via in-line GC or a continuous monitor; or exhaust gas will be collected in Tedlar® bags for off-line 20 analysis of CO. Tedlar® bag samples may be collected at 21 the exit of the bubblers described in Section C.2.4.6. 22 23 C.2.4.6 Exhaust Gas Sampling 24 25 Gas samples for off-line analysis will be collected as 26 described in Appendix D.1, revised as necessary pursuant to 27 Appendix C.1.2.2 if applicable. 28 29 30 A minimum of 60 mL of bubbler aqueous solution composite is expected from each combustion test. Of this, a minimum of 31 32 45 mL will be directed to PFOA analysis, and the remainder will be directed to fluoride ion analysis. 33 34 35 C.2.4.7 Exhaust Gas Analysis 36 37 C.2.4.7.1 Fluoride Ion 38 39 A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section 40 C.2.4.6 will be analyzed for fluoride ion via ion 41 42 chromatography using EPA Method 300.0. 43 C.2.4.7.2 PFOA 44 45 46 A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section 47

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C.2.4.6 will be analyzed for PFOA via LC/MS/MS as described 1 2 in Appendix D.2. 3 4 As described in Appendix D.2, composite bubbler aqueous 5 solution sample results less than method detection limit 6 (MDL) will be reported as not detected (ND), results 7 between MDL and the limit of quantitation (LOQ) will be 8 reported as not quantifiable (NQ), and numerical values 9 will not be reported. 10 Due to background levels of PFOA, the analytical laboratory 11 will only report numerical values for PFOA concentration in 12 13 the aqueous solution greater than or equal to the LOQ. This is required to assure that the reported concentration 14 value is attributable to the aqueous solution sample rather 15 16 than to background. 17 C.2.5 Reporting of Results 18 19 20 C.2.5.1 Elemental Analysis Results 21 The results of elemental analysis for each test substance 22 composite (as noted in Section C.2.1) will be reported. 23 24 The laboratory reports will be included in an appendix to 25 the final report for incineration testing (test report). 26 27 C.2.5.2 Combustion Stoichiometry Results 28 Combustion stoichiometry (as noted in Section C.2.2) 29 30 calculations for each test substance composite will be 31 included in an appendix to the test report. 32 33 C.2.5.3 TGA Results 34 35 The temperature for complete gasification and the TGA 36 graphical results for each test substance composite (as noted in Section C.2.3) will be included in an appendix to 37 38 the test report. 39 Combustion Test Results C.2.5.4 40 41 C.2.5.4.1 Process Monitoring 42 43 Process monitoring data (as noted in Section C.2.4.4) 44 45 recorded for each combustion test will be reported in 46 tabular form. 47

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C.2.5.4.2 Exhaust Gas Monitoring 1 2 Exhaust gas O_2 , CO and CO_2 monitoring results will be 3 reported as the integrated or average value for each 4 5 combustion test. CO will be reported in terms of parts per million by volume (ppmv). O_2 and CO_2 will be reported in 6 7 terms of percent by volume (%). 8 9 C.2.5.4.3 Exhaust Gas Analytical Results 10 11 Results of analyses noted in Section C.2.4.7 will be reported for each replicate of each combustion test. 12 13 14 The analytical result for each analyte in Section C.2.4.7 will be reported in terms of concentration (mass per 15 16 volume) in the bubbler aqueous solution. For each analyte, 17 this value will be used with the associated exhaust gas volume to compute an exhaust gas concentration and with the 18 associated test substance mass to compute mass of analyte 19 per mass of test substance composite. 20 21 22 C.2.5.4.3.1 Fluoride 23 24 Fluoride ion in the exhaust gas will be reported on the basis of mass of fluoride ion per mass of test substance 25 composite. The corresponding hydrogen fluoride value for 26 each will also be computed and reported for reference. 27 28 29 C.2.5.4.3.2 PFOA 30 PFOA results for the bubbler aqueous solution samples will 31 be reported as described in Section C.2.4.7.2. PFOA 32 results for associated blanks will also be reported. 33 34 If present in the bubbler aqueous solution at a 35 concentration above the matrix-specific LOQ, PFOA in the 36 exhaust gas will be reported on the basis of mass of PFOA 37 38 per mass of test substance composite. 39 C.2.5.5 Release Assessment 40 41 In the event that PFOA is reported for the exhaust gas 42 bubbler aqueous solution at a concentration at or above the 43 LOQ (as defined in Appendix D.2) for two or more of the 44 45 three runs for a given test substance composite, a release assessment report for the full-scale waste incineration of 46 products represented by the test substance composite will 47

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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

9 applicable.

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1 APPENDIX D.1

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2 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.

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

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

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1 APPENDIX D.2 2 PFOA ANALYSIS BY LC/MS/MS 3 4 D.2.1 Introduction 5 6 Samples to be analyzed for PFOA in this study will be 7 subjected to Liquid Chromatography with Tandem Mass 8 Spectrometry (LC/MS/MS) in accordance with "Method of 9 Analysis for the Determination of Ammonium 10 Perfluorooctanoate (APFO) in Water Revision 1" (Exygen method) revised per the section-by-section comments listed 11 12 in Section D.2.4 below. These revisions are necessary to adapt a method originally developed for liter quantity 13 14 water samples to samples related to testing described in 15 Appendix C. 16 17 The testing programs described in Appendix C are expected to generate samples of aqueous solution, methanol (e.g., as 18 used for extraction or rinsing), and corresponding blanks. 19 The expected sample size for aqueous solution samples (from 20 21 exhaust gas bubbler sample collection) available for 22 analysis via this method is approximately 50 mL. 23 24 .D.2.2 Method Summary 25 26 PFOA is extracted from water using a disposable C_{18} solid 27 phase extraction (SPE) cartridge. PFOA is eluted from the cartridge with methanol. Quantification of PFOA is 28 accomplished by electrospray liquid chromatography/tandem 29 mass spectrometry (LC/MS/MS) analysis. 30 31 32 D.2.3 Reporting 33 The target limit of quantitation (LOQ) for this study with 34 this method is 50 ng/L based on prior work with water 35 36 samples where an 8-fold concentration via extraction using 37 C_{18} SPE cartridge has been demonstrated. The actual LOQ 38 will be matrix dependent; for samples (e.g., methanol rinsate) where the 8-fold concentration cannot be 39 performed, the target LOQ for this study is 400 ng/L. 40 41 42 Sections 4.5.4 and 5 of the Exygen method explain reporting for field samples such as bubbler aqueous solution 43 composites, which are distinct from blanks and spikes, as 44 45 follows: 46 Field samples in which either no peaks or peaks 47

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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).

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8 Therefore, sample results less than method detection limit (MDL) will be reported as ND, and sample results between 9 MDL and the limit of quantitation (LOQ) will be reported as 10 NQ. Numerical values will not be reported for such 11 samples. Only concentrations above the LOQ, where the 12 reported concentration is attributable to the sample rather 13 14 than to background, are reported with numerical values.

16 Additionally, if the PFOA anion is found in a sample at a 17 concentration above the LOQ for the matrix but is less than 5 times the concentration found in the associated blank, 18 the result will be flagged and treated as ND. 19

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D.2.4 Study-Specific Comments on the Method

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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 C₁₈ SPE cartridge described in section 4.4 of the method, the LOQ and MDL in the method will be a factor of 8 higher than reported where less than 40 mL of sample is available or where the sample is not amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of the method will be a factor of 8 higher than reported where less than 40 mL of sample is available or where the sample is not amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of the method (e.g., methanol).
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.

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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 C₁₈ SPE cartridge described in section 4.4 of the method, then section 4.4 is skipped such that the sample is analyzed directly. (Note: For such samples, the LOQ and MDL will be 8 times higher than 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 C₁₈ SPE cartridge requires a suitable aqueous sample. This extraction and the corresponding 8-fold concentration

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	pointed out in the NOTE at the end of this section cannot be performed on non-aqueous (e.g., methanol) samples.
4.4, item 1	• 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.

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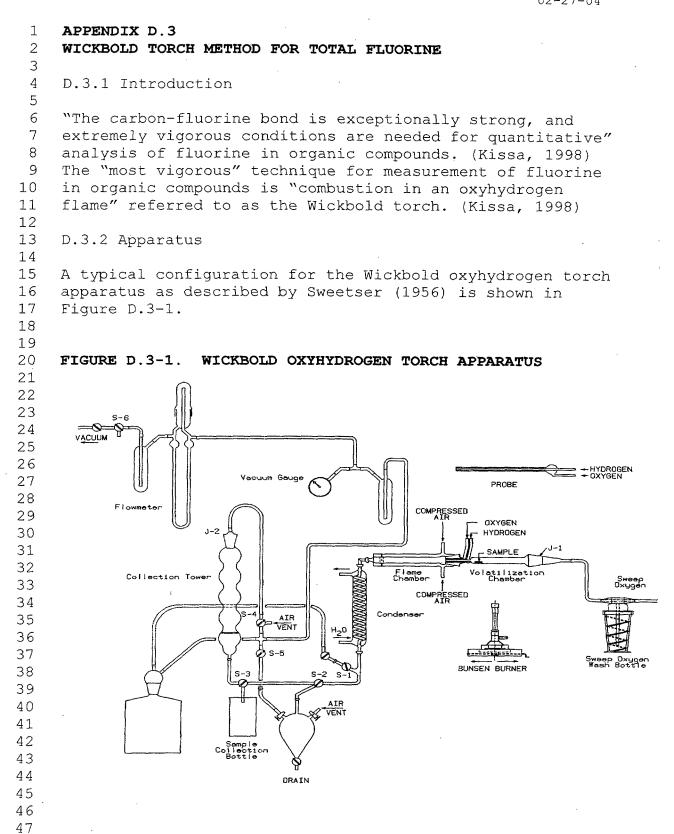
D.2.5 Reference

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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
January 2003. (EPA Docket ID OPPT-2003-0012-0040)

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1 D.3.3 Method Description 2 3 The sample size for the standard sample boat is up to 20 mg 4 for a solid or up to 5 mL for a liquid. 5 б With the oxyhydrogen torch in operation, the sample is 7 pyrolyzed or vaporized with a Bunsen burner moving on a 8 rail below the volatilization chamber. The vapors and pyrolysis products are swept through the oxygen-hydrogen 9 10 flame chamber operating at up to approximately 2000 °C to 11 mineralize the fluorine in the sample to fluoride ion. The 12 resulting fluoride ion is absorbed in the collection tower 13 containing water or an alkaline solution. 14 15 The absorbed fluoride ion is measured via fluoride ion-16 selective electrode or ion chromatography. 17 18 The reported limit of quantitation for total fluorine via 19 the Wickbold Torch method is 0.5 ppm (0.5 mg/kg). The 20 accuracy of this method for determination of total fluorine in fluorinated polymers is exemplified by total fluorine 21 22 values of 75.35% to 75.84% for PTFE with known total 23 fluorine content of 76.0%. (Sweetser, 1956) 24 25 D.3.4 Safety Considerations 26 27 Use of hydrogen presents a potential fire and explosion 28 hazard. Use of oxygen presents a potential fire hazard. 29 Safe operation of the oxyhydrogen torch is assured by the 30 use of specialized equipment with shielding and elaborate safety devices by well-trained personnel at a qualified 31 32 laboratory. 33 D.3.5 References 34 35 "Analysis of Anionic Fluorinated Surfactants", 36 Kissa, E. 37 Chapter 8 in Anionic Surfactants: Analytical Chemistry -38 2nd Edition, Revised and Expanded, edited by John Cross. 39 Marcel Dekker Surfactant Science Series, volume 73, 1998. 40 Sweetser, P. B. "Decomposition of Organic Fluorine 41 42 Compounds by Wickbold Oxyhydrogen Flame Combustion Method", 43 Analytical Chemistry, vol. 28, pp. 1766-1768, 1956.

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1 APENDIX D.4

2 WASTE INCINERATION AND OPERATION CONDITIONS

4 Polymers of the sort being investigated in this testing 5 program may be present at trace to low concentrations in 6 the feedstreams to municipal waste combustors and/or 7 medical waste incinerators in the U.S.

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D.4.1 Types of Incinerators

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11 D.4.2.1 Municipal Waste Combustors

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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.

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20 Table D.4-1. MWCs in 2002

		A A A A A A A A A A A A A A A A A A A	-	
Туре	Number of	Annual Capacity	Fraction	
	Facilities	(million Ton/year)	of Waste	
Mass Burn	68	22.5	76.5%	
Refused Derived	18	6.4	21.8%	
Fuel (RDF)				
Modular	. 12	0.5	1.7,8	
Total	98	29.4	100.0%	

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22 D.4.1.2 Hospital/Medical/Infectious Waste Incinerators 23

24 Although earlier reports indicated approximately 2400 25 medical waste incinerators in the U.S. in the 1990s burning approximately 846 thousand tons of hospital and 26 27 medical/infectious waste (EPA 1997), the current EPA Office of Air Quality, Planning, and Standards (OAQPS) inventory 28 29 indicates that there are 116 hospital/medical/infectious 30 waste incinerators (HMIWIS) in the U.S. as of July 28, 2003. (EPA 2003) 31

33 This represents a greater than 90% reduction in the number of operating HMIWIs in the U.S. Many medical waste 34 35 _ incinerators were closed rather than upgraded to meet new emission standards, as hospitals improved their programs to 36 37 segregate infectious ("red bag") waste burned in HMIWIs 38 from non-infectious ("black bag") waste handled as 39 municipal solid waste after it leaves the hospital. 40 Consequently, the amount of segregated infectious waste

1 burned in HMIWIs is expected to be less than 0.3 million 2 tons per year. 3 EPA notes that over 97% of medical waste incinerators are 4 5 controlled air modular units (EPA 2000a). Recent communication with EPA OAQPS indicates that virtually all 6 7 existing HMIWIs are controlled air modular (two-chamber) units. 8 9 10 D.4.2 Incinerator Operating Conditions 11 12 Many incinerators for municipal solid waste are designed to operate in the combustion zone at 1800 °F (982 °C) to 2000 13 °F (1093 °C) to ensure good combustion. (EPA 1995) EPA's 14 new source performance standards (NSPS) and emission 15 guidelines for both municipal waste combustors (MWCs) and 16 hospital/medical/infectious waste incinerators (HMIWIs) are 17 based on the use of "good combustion practices" (GCP). (EPA 18 1997, EPA 2000b, EPA 2000c, Van Remmen 1998) 19 20 Referring to MWCs, Donnelly notes, "Design of modern 21 efficient combustors is such that there is adequate 22 23 turbulence in the flue gas to ensure good mixing, a hightemperature zone (greater than 1000 °C) to complete burnout, 24 and long enough residence time at high temperature (1-2 25 sec) for complete burnout." (Donnelly 2000) The term "flue 26 27 gas" here refers to the gas above the grate. 28 29 With respect to HMIWIS, Van Remmen states "any unit which presently [prior to compliance date] has a [secondary 30 chamber] residence time less than two seconds at 1000 °C 31 does not meet the requirement for good combustion under the 32 new regulations." (Van Remmen 1998) 33 34 35 Similarly, most MWCs operate with a 2 second gas residence time in the high temperature zone in order to assure 36 37 compliance with emission standards on carbon monoxide (CO) 38 and dioxins. 39 40 D.4.2.1 MWC Operating Conditions 41 42 D.4.2.1.1 Mass Burn MWC 43 Review of the IWSA Directory (IWSA 2002) indicates that 44 almost all of these mass burn units are mass burn water 45 wall furnaces. Nearly all mass burn water wall furnaces 46

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1 have reciprocating grates or roller grates to move the 2 waste through the combustion chamber. (EPA 1996a) 3

4 Studies on the Millbury, Massachusetts mass burn water wall 5 MWC produced gas temperature versus residence time results. 6 (Scavuzzo, Strempek, and Strach 1990) Calculations based 7 on Figure 6 of this paper indicate a time-averaged 8 temperature of 2238 °F (1226 °C) over a 2 second. The 9 corresponding gas temperature at the 2 second level from 10 this figure is 1750 °F (954 °C).

12 A report on the Warren County, New Jersey mass burn water 13 wall MWC indicates that the design gas temperature between the grate and secondary air inject was greater than 2000 $^\circ {\rm F}$ 14 15 (1093 °C) over a gas residence time of an additional 2.2 seconds. (Schuetzenduebel and Nobles 1990) This report 16 17 also shows that this MWC was designed for 2 seconds 18 residence time above 1800 °F (982 °C) between the 19 introduction of secondary air and the exit of the furnace section. (Schuetzenduebel and Nobles 1990) The temperature 20 21 profile (Figure 21) in the temperature correlation test report (Schutzenduebel 1989) for this MWC shows the full 22 load gas temperature at the secondary air injection point . 23 is 2650 $^{\circ}$ F, and the gas temperature at the 2-second point is 24 25 1850 °F. Therefore, testing indicates an average temperature of 2250 °F (1232 °C) over this 2 second gas 26 residence time for the Warren County unit. A related 27 report for the Warren County MWC by the design firm 28 29 indicates that the exhaust gas oxygen concentration is nominally 10% (dry basis). (Blount Energy Resource Corp. 30 31 1989) 32

33 Information from these 2 MWCs demonstrates that the average 34 gas temperature across a 2 second residence time for mass 35 burn MWCs is conservatively expected to be greater than 36 1100 °C.

38 Test report data from a typical mass burn MWC (Fairfax, 39 Virginia) indicates typical average furnace exit gas 40 concentrations are 10.8% oxygen (dry basis) and 18.4% 41 moisture (water). (Clean Air Engineering, 1997)

As indicated in Table D.4.1, mass burn units account for 44 over 76% of the municipal solid waste incinerated in the 45 U.S.

2 D.4.2.1.2 RDF MWC

4 Furnace temperatures as well as flue gas oxygen and moisture 5 (H₂O) levels for the Mid-Connecticut RDF combustor during 6 performance tests while operating under good combustion 7 conditions across a range of steam loads (Finklestein and 8 Klicius 1994) are summarized in Table D.4-2.

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10 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

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The average operating conditions for this RDF unit across the range of steam loads are 1016 °C, 8.4% O₂ (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 23 the U.S. involves burning on the grate (EPA 1996a) similar 24 to the operation of mass burn MWCs, the time-temperature 25 26 profile in an RDF unit is expected to be similar to that 27 described in Section D.4.2.1.1 above. Based on this similarity and the temperatures in Table D.4-2, the average 28 gas temperature across a 2 second residence time for RDF 29 units is conservatively expected to be greater than 1100 °C. 30 31

32 As indicated in Table D.4.1, RDF units account for 33 approximately 22% of the municipal solid waste incinerated 34 in the U.S.

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D.4.2.1.1 Modular MWC

38 Modular MWCs are generally small dual-chamber units, 39 accounting for less than a total of 2% of the municipal 40 solid waste incinerated in the U.S. in 2002. Modular MWCs 41 are generally equipped with auxiliary fuel burners in the

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secondary chamber. (EPA 1996a) EPA notes that the 1 2 secondary chamber exit temperature of modular MWCs is 3 maintained at typically 980 to 1200 °C. (EPA 1996a) 4 5 A typical modular MWC in Polk County, Minnesota is operated 6 with a gas residence time of 2 seconds, in the secondary 7 chamber, a secondary chamber exit temperature in the range of 1800 °F (982 °C) to 2000 °F (1093 °C), flue gas oxygen 8 concentrations in the range of 10% to 13% (dry basis), and 9 10 flue gas moisture in the range of 10% to 15% (Pace 11 Analytical 2003). 12 13 Since the secondary chamber exit temperature is expected to be the minimum gas-phase temperature for the chamber, the 14 secondary chamber average gas temperatures for modular MWCs 15 16 are expected to be 1000 °C or greater. 17 18 As indicated in section D.4.1, such modular units are 19 generally small MWCs and account for less than a total of 2% of the municipal solid waste incinerated in the U.S. 20 21 22 D.4.2.1.4 MWC Summary 23 Considering the relative quantities of municipal waste 24 burned annually in each type of MWC and the data in this 25 26 section, typical operating conditions for the high 27 temperature zone of most MWCs are >1000 °C average 28 temperature across 2 second residence time with exit gas 29 concentrations of 10% O_2 (dry basis) and >15% moisture. 30 31 D.4.2.2 HMIWI Operating Conditions 32 33 The range of temperatures for the secondary chamber of controlled air medical waste incinerators has been reported 34 as 980 to 1200 °C. (Theodore 1990) EPA notes that auxiliary 35 fuel (e.g., natural gas) is burned in the secondary chamber 36 37 of medical waste incinerators to sustain temperatures in the range of 985 to 1095 $^{\circ}\mathrm{C}$ and that combustion air at 150 38 to 250 % of the stoichiometric requirement is usually added 39 40 to the secondary chamber. (EPA 2000a, EPA 1994a) 41 In its model plant description background document, EPA 42 43 notes that the average moisture content in HMIWI flue gas was about 10 % based on available data, and EPA states 44 "limited data show that older [HMIWI] units typically have 45 residence times that range from essentially 0 seconds up to 46 47 about 1 second." (EPA 1994b) However, as noted above, a

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more recent report indicates that HMIWIs still in operation 1 have secondary chamber temperatures greater than or equal 2 to 1000 °C with a gas residence time of 2 seconds. (Van 3 Remmen 1998) For example, EPA studied the incinerator at 4 5 Weeks Hospital in New Hampshire as a typical HMIWI with a design residence time of 2 seconds in the secondary 6 chamber. (EPA 1996b) During this testing, the average exit 7 secondary chamber exit temperature was 1024 °C, and the flue 8 9 gas oxygen concentration was 13.5%. (dry basis) (EPA 1996b) 10 Review of test reports for all HMIWIS in the EPA docket for 11 the HMIWI NSPS and EG rulemakings that are listed in EPA's 12 current HMIWI inventory (EPA 2003) does not refute Van 13 14 Remmen's statement above on residence time and temperature and indicates HMIWI flue gas oxygen concentrations for 15 these units in the range of 10 to 15% (dry basis) and stack 16 moisture concentrations as high as 30% (after wet 17 (Environmental Laboratories Inc. 1993, EPA 18 scrubbing). 19 1996, HDR Engineering 1994a, HDR Engineering 1994b, METCO Environmental 1992, Technical Services, Inc. 1993, 20 Technical Services, Inc. 1994a, Technical Services, Inc. 21 Apparently, the older HMIWIs referred to in EPA's 22 1994b) model plant description background document either have 23 been shut down or upgraded to operate with secondary 24 chamber exit temperatures higher than 1000 °C at a gas 25 residence time of 2 seconds. 26 27 Secondary chamber temperature of HMIWIs is monitored near 28 the secondary chamber outlet. (EPA 1994) Hence, when the 29 auxiliary burner (located on the end opposite from the 30 31 outlet) is in use, the average gas temperature in an HMIWI secondary chamber is greater than the outlet temperatures 32 noted above. Therefore, secondary chamber average gas 33 temperatures for HMIWIs are expected to be 1000 °C or 34 greater with a gas residence time of 2 seconds. 35 36 In summary, typical operating conditions for the secondary 37 chamber of operating HMIWIs in the U.S. are 1000 °C average 38 temperature across 2 second residence time with exit gas 39 concentrations of 13% O_2 (dry basis) and >10% moisture. 40 41 D.4.3 Pollution Control Equipment 42 43 Over 99% of large MWC capacity operates with a spray dryer 44absorber/scrubber. (IWSA 2003) Approximately 80% of large 45 MWC capacity operates using carbon injection as part of the 46 pollution control system. (IWSA 2003) Due to requirements 47

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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.

7 Review of EPA's HMIWI inventory (EPA 2003) indicates that 8 essentially all HMIWIs have some form of wet or dry 9 scrubbing for acid gas control.

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11 D.4.4 Summary

13 Approximately 30 million tons per year of municipal solid waste was combusted in the United States annually in waste-14 15 to-energy municipal waste combustors in 2003. 16 Approximately 0.3 million tons per year of segregated 17 medical waste was combusted annually in the United States 18 in hospital/medical/infectious waste incinerators in 2003. Considering the relative amounts of waste combusted 19 20 annually, typical operating conditions for waste incineration in the U.S. across these two classes of units 21 are as follows: 22

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

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EPA emission regulations currently in place or in place by 26 2005 require that operating municipal waste combustors and 27 hospital/medical/infectious waste incinerators have or will 28 have air pollution control equipment such as wet or dry 29 scrubbing for acid gas control. 30

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1 References

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3 Blount Energy Resource Corp. Correlation Procedure for 4 Continuously Monitoring Furnace Temperatures (Warren County 5 Resource Recovery Facility), March 22, 1989. 6 Clean Air Engineering. Test Report for Covanta of Fairfax, 7 Inc. I-95 Energy/Resource Recovery Facility, 1997. 8 9 Donnelly, J.R. Waste Incineration Sources: Municipal Waste 10 Combustion. In: W.T., ed., Air Pollution Engineering 11 Manual, 2nd edition. Air and Waste Management Association. 12 13 New York, NY: Van Nostrand Reinhold, 2000, pp 257-268. 14 15 Environmental Laboratories Inc. Stack Test Report for 16 Emissions Testing of the Bethesda Memorial Hospital Waste 17 Incinerator, Boynton Beach Florida, September 13, 1993. 18 19 Environmental Protection Agency (EPA). Municipal Waste Combustion Assessment: Technical Basis for Good Combustion 20 21 Practice, EPA 600/8-89-063, August 1989. 22 23 EPA. Medical Waste Incinerators - Background Information for Proposed Standards and Guidelines: Control Technology 24 Performance Report for New and Existing Facilities, EPA-25 26 453/R-94-044a, July 1994. 27 28 Medical Waste Incinerators - Background Information EPA. for Proposed Standards and Guidelines: Model Plant 29 30 Description and Cost Report for New and Existing 31 Facilities, EPA-453/R-94-045a, July 1994. 32 33 Decision Maker's Guide to Solid Waste Management, EPA. 34 Volume II, Chapter 8, 1995. 35 EPA. AP-42, Fifth Edition, Volume I, Chapter 2: Solid 36 37 Waste Disposal, Section 2.1, Refuse Combustion, Supplement 38 B, October 1996. 39 EPA. Medical Waste Incineration Emission Test Report: 40 41 Weeks Memorial Hospital, Lancaster, New Hampshire, EMC 42 Report 96-MWI-11, March 1996. 43 44 Standards of Performance for New Stationary Sources EPA. 45 and Emission Guidelines for Existing Sources: Hospital/Medical/Infectious Waste Incinerators, 62 Federal 46 47 Register 48346, September 15, 1997.

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APPENDIX E.1 OUTLINE FOR INTERIM PROGRESS REPORTING Title: Enforceable Consent Agreement for the Laboratory-Scale Incineration Testing of Fluorotelomer Based Polymers - Interim Report OPPT Docket ID No: OPPT-2004-0001 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: Description of Difficulties: (If none indicate N/A) 2) 3) Actions taken in response to difficulties: If none indicate N/A) Other information relevant to the progress of the 4) testing program: (If none indicate N/A)

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1 APPENDIX E.1 (continued)		
2	OUTL	INE FOR INTERIM PROGRESS REPORTING
3		
4 5 6 7	Title	e: Enforceable Consent Agreement for the Laboratory- Scale Incineration Testing of Fluoropolymers - Interim Report
8 9	OPPT	Docket ID No: OPPT-2003-0071
10		
11 12 13	Date	of Interim Report: [date]
14 15 16 17	This	Report covers the period from [date] to [date]
18 19 20 21 22 23	1)	List or description of significant ECA Test Program milestones during this period:
24 25 26 27 28 29 30	2)	Description of Difficulties: (If none indicate N/A)
31 32 33 34 35 36 37 38 39	3)	Actions taken in response to difficulties: If none indicate N/A)
40 41 42	4)	Other information relevant to the progress of the testing program: (If none indicate N/A)

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1 APPENDIX E.2

2 OUTLINE FOR RELEASE ASSESSMENT REPORT

As described in Appendix C.2.5.5 of this ECA, if PFOA is 4 reported for the exhaust gas bubbler aqueous solution at a 5 concentration at or above the LOQ (as defined in Appendix 6 D.2) for two or more of the three runs for a given test 7 substance composite, then the potential for release from 8 full-scale municipal and/or medical waste incineration, as 9 applicable, (including application of air pollution 10 controls) of products represented by the test substance 11 composite in the United States will be assessed to put the 12 data into perspective. At a minimum, the report will 13 follow the general outline described below and will state 14 assumptions, document the basis for the assumptions made, 15 quantitatively estimate the variability of calculated 16 estimates (based on the variability of the parameters in 17 the evaluation), and qualitatively discuss the uncertainty 18 of calculated estimates. 19

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1.0 Introduction

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37 38 • 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.

31 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.
- 39 40

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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

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1 2 3 4		operational parameters. Cross-reference to or submission of relevant parts of Appendix D.4 of this ECA can satisfy this provision.
5 6 7 8 9		• 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.
10 11 12 13 14	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.
15 16 17		• The relevance of the subject test substance composite to MWCs and/or medical waste incinerators.
18 19 20 21 22 23 24		• 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.
25 26	,	• A description of the extrapolation.
27 28		• A description of any assumptions used.
29 30 31 32 33	·	 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.
34 35	5.0	Sensitivity Analysis
36 37 38 39		 Assessment of the impact of variability (quantitative) and uncertainty (qualitative) in each parameter on the evaluation results.
40 41	6.0	Conclusions
42	7.0	References

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APPENDIX E.3 1 2 OUTLINE OF TEST REPORT*',** 3 4 5 Table of Contents 6 7 Executive Summary 8 9 Introduction 10 I. Phase I PFOA Transport Testing 11 12 13 1. Experimental Apparatus 2. Description of Test Conditions (including deviations 14 15 from protocol) 16 3. Documentation of PFOA Standard 17 4. Analytical Results 18 4.1 PFOA 19 4.2 Total Fluorine 20 5. Transport Efficiency 21 5.1 PFOA 22 5.2 Total Fluorine 23 6. Discussion of Results 7. Conclusions 24 25 II. Phase II Incineration Testing (provided Phase II is performed) 26 27 28 1. Documentation of Test Substance Composites 29 2. Elemental Analysis Results 3. Combustion Stoichiometry Results 30 31 4. TGA Results 32 5. Combustion Testing 33 5.1 Experimental Apparatus 5.2 Description of Test Conditions (including deviations 34 35 from protocol) 36 5.3 Combustion Testing Results 37 5.3.1 Process Monitoring 38 5.3.2 Exhaust Gas Monitoring 39 5.3.2 Exhaust Gas Sampling and Analysis 40 5.3.2.1 PFOA 41 5.3.2.2 Fluoride 42 5.4 Discussion of Results 43 5.5 Conclusions 44 45 III. Appendices 46 47 • Quality Assurance Report(s)

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1		·
2		• Report(s) from Compositing Facility(ies) (provided Phase II
3		is performed)
4		
5		 Reports from Analytical Laboratories
6		•
7		 Release Assessment per Appendix E.2, if applicable
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10		
11	*	Test Report will include this information (as applicable)
12		but not necessarily in this format.
13		,
14	**	References to literature in this report will include full
15		citations.

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1 APPENDIX F

2 ECA INCINERATION TESTING QUALITY ASSURANCE PROJECT PLAN (QAPP):

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- 3 REQUIRED CONTENT
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EPA QA/R-5 QAPP Guidance Element	Required Content of QAPP(s) for ECA Incineration Testing
A: PROJECT MANAGEMENT	
A1 Title and Approval Sheet	to be included in QAPP
A2 Table of Contents	to be included in QAPP
A3 Distribution List	to be included in QAPP
A4 Project/Task Organization	to be included in QAPP
A5 Problem Definition/ Background	to be satisfied by cross- reference to ECA (Parts I, IV) and Appendix A, C.1, or C.2, as applicable
A6 Project/Task Description	see element A5
A7 Quality Objectives and Criteria	to be satisfied by cross- reference to Appendix A, C.1, or C.2 (as applicable) and to Appendix D.2 and/or D.3, as applicable
A8 Special Training/	for facilities subject to
Certifications	GLP (40 CFR Part 792) under this ECA, QAPP shall state that this element is satisfied by compliance with applicable GLP requirements; for compositing facilities, to be to be satisfied by providing a statement of the qualifications for each such facility
A9 Documentation and Records	to be satisfied by cross- reference to ECA Part XIV and Appendix E
B: DATA GENERATION AND ACQUISITION	
B1 Sampling Process Design (Experimental Design)	see element A5
B2 Sampling Methods	to be satisfied by cross- reference to Appendix C.1 or C.2 (as applicable) and to Appendix D.1
B3 Sample Handling and Custody	to be included in QAPP consistent with Appendix A
B4 Analytical Methods	to be satisfied by cross-

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	reference to analytical
	method descriptions in
	Appendices C.2, D.2, and
	D.3, as applicable
B5 Quality Control	to be satisfied by cross-
	reference to QC provisions
	(e.g., blanks) in
	Appendices A, C.1, C.2,
	D.2, and D.3, as applicable
P6 Instrument /Fauipment	to be included in QAPP in
B6 Instrument/Equipment	
Testing, Inspection, and	summary form for chemical
Maintenance	analysis equipment for the
	analytical methods for
	element B4 above
B7 Instrument/Equipment	to be included in QAPP in
Calibration and Frequency	summary form for chemical
	analysis equipment for the
	analytical methods for
	element B4 above
B8 Inspection/Acceptance of	for facilities subject to
Supplies and Consumables	GLP (40 CFR Part 792) under
Suppries and consumpties	this ECA, QAPP shall state
	that this element is
	satisfied by compliance
	with applicable GLP
	requirements; not
	applicable to other
	facilities
B9 Non-direct Measurements	to be satisfied by cross-
	reference to Appendix C.2.2
B10 Data Management	for facilities subject to
	GLP (40 CFR Part 792) under
	this ECA, QAPP shall state
	that this element is
	satisfied by compliance
	with applicable GLP
	requirements; for
	compositing facilities, to
	be to be satisfied by
	cross-reference to Appendix
	A.4
C: ASSESSMENT AND OVERSIGHT	
C1 Assessments and Response	for facilities subject to
Actions	GLP (40 CFR Part 792) under
	this ECA, QAPP shall state
	that this element is
	satisfied by compliance
	DUCTOTIED DY COMPTIANCE

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	with applicable GLP
	requirements; for other
	facilities, to be included
``````````````````````````````````````	in QAPP
C2 Reports to Management	for facilities subject to
	GLP (40 CFR Part 792) under
	this ECA, QAPP shall state
	that this element is
	satisfied by compliance
	with applicable GLP
	requirements; for other
	facilities, to be included
	in QAPP
D: DATA VALIDATION AND USABILITY	
D1 Data Review, Verification,	to be satisfied by cross-
and Validation	reference to Appendix A,
and varidation	
	C.1, or C.2 (as applicable)
	and to Appendix D.2 and/or
	D.3, as applicable
D2 Verification and	for facilities subject to
Validation Methods	GLP (40 CFR Part 792) under
	this ECA, QAPP shall state
	that this element is
	satisfied by compliance
	with applicable GLP
	requirements; for other
	facilities, to be included
	in QAPP consistent with
	Appendices A, C.1, C.2,
	D.2, D.3 as applicable
D3 Reconciliation with User	to be satisfied by cross-
Requirements	reference to Appendices
- ,	C.2.5.5 and E.2, as
	applicable

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1	APPENDIX G
2	COPY OF EPA ORDER
3	
4	
5	UNITED STATES
6	ENVIRONMENTAL PROTECTION AGENCY
7	CONTRACTOR TOD TOD TADODATONY CONTRACTOR
8	TESTING CONSENT ORDER FOR THE LABORATORY-SCALE INCINERATION TESTING OF FLUOROTELOMER BASED POLYMERS
9	TESTING OF FLOOROTELOMER BASED FOUTHERS
10	Docket No. OPPT-2004-0001
11	DOCKEE NO. OIII 2004 0002
12 13	
14	Under the authority of section 4 of the Toxic Substances
15	Control Act (TSCA), 15 U.S.C. 2603, the United States
16	Environmental Protection Agency (EPA) issues this testing
17	consent order (Order) to take effect on the date of publication
18	of the notice in the Federal Register announcing the issuance of
19	this Order. This Order incorporates the enforceable consent
20	agreement (ECA) for the laboratory-scale incineration testing of
21	fluorotelomer based polymer test substance composites listed in
22	Appendix A of the ECA.
23	
24	

30	
31 Date	Stephen L. Johnson,
32	Assistant Administrator
33	For Prevention, Pesticides,
34	And Toxic Substances
35	

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## 1 APPENDIX G (continued) 2 COPY OF EPA ORDER

### UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

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

Docket No. OPPT-2003-0071

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

Date	Stephen L. Johnson,
	Assistant Administrator For Prevention, Pesticides, And Toxic Substances

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