1 APPENDIX B.1

2 GUIDELINE FOR THERMOGRAVIMETRIC ANALYSIS

3

ASTM E 1868-02 "Standard Test Method for Loss-On-Drying by 4 5 Thermogravimetry" will be used as the guideline for conducting the analysis described in Appendix C.2.3 with the following 6 7 modifications for this testing program:

8

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

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

9 10

11 Reference

12 ASTM E 1868-02 "Standard Test Method for Loss-On-Drying by

13 Thermogravimetry", ASTM International. For referenced ASTM

14 standards, visit the ASTM website, www.astm.org, or contact ASTM

15 Customer Service at service@astm.org. For Annual Book of ASTM

16 Standards volume information, refer to the standard's Document

17 Summary page on the ASTM website.

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

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

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 4 3) and/or total fluorine (equation 4) would be 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 $mass_{\text{PFOA}}$ 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 mass of PFOA fed to thermal and $mass_{PFOA}$ in = 18 reactor system 19 20 mass_{total F} out 21 % Total F TE = ----- * 100 (4) 22 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 and mass_{total F} in = calculated mass of 31 total F in PFOA fed to 32 thermal reactor system 33 34 Step 2 (if necessary) The experiment described in Section 35 C.1.2.1 would be repeated with 36 reagent(s) (e.g. NaOH) added to the 37 bubbler aqueous solution to determine 38 if reagent addition enhances analyte absorption, thereby improving transport 39 40 efficiency. Transport efficiency would 41 be calculated using equation (1) and/or 42 (2) above. The impact of reagent 43 addition on LOQ for PFOA analysis 44 described in Appendix D.2 would be 45 determined. 46 47 C.1.3 Reporting of Results 48 49 Following completion of PFOA transport testing as described 50 in this appendix and prior to beginning incineration testing described in Appendix C.2, a letter report will be 51

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

4

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.

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

1 will be considered to be the same as the total fluorine 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 11 content of each test substance composite is needed. Based on manufacturing process knowledge of the polymers in this 12 13 program, levels of sulfur, and nitrogen are expected to be 14 less than 0.1% and to thereby have negligible effect on 15 stoichiometric calculations. 16 17 C.2.1.3.1 Theoretical Determination 18 19 Where the elemental composition of a test substance 20 composite is known from the identity of the components in a given composite, the carbon and hydrogen content of the 21 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 | | | 100 |

30 C.2.1.3.2 Empirical Determination

31

29

32 Where compositional information on carbon and hydrogen 33 content is not known from the identity of the components in 34 a given composite, each such test substance composite will 35 be analyzed for carbon and hydrogen.

36

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

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

1 representative sample is weighed and placed in a crucible (or dish) and evaporated to dryness in an air or nitrogen 2 3 atmosphere at a defined temperature setpoint (e.g., 103 °C to 105 °C) in the range of 100 °C to 125 °C. The moisture 4 5 value is calculated as the loss in weight (difference between the starting weight of sample and the final weight 6 7 of sample) divided by the starting weight of sample. 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 14 Section C.2.2 will be performed to aid in preparation for 15 combustion testing described in Section C.2.4 16 17 First, the weight percent values from Section C.2.1 are 18 converted to molar quantities on a dry basis. 19 20 Second, based on Chapter 3 of Combustion Fundamentals for Waste Incineration (American Society of Mechanical 21 22 Engineers, 1974), the reaction products for these molar 23 quantities are calculated assuming complete combustion with 24 the following rules: 25 26 a) All carbon (C) in feed converts to carbon dioxide (CO_2) 27 $C + O_2 \rightarrow CO_2$ 28 b) All sulfur (S) in feed converts to sulfur dioxide (SO₂) 29 30 $S + O_2 \rightarrow SO_2$ 31 32 c) The halogens (Cl, F) in feed convert to hydrogen halides $H_2 + Cl_2 \rightarrow 2HCl$ 33 34 $H_2 + F_2 \rightarrow 2HF$ 35 36 d) Hydrogen (H) present in feed in excess of that 37 required to yield products in item c) above will be 38 converted to water 39 $2H_2 + O_2 \rightarrow 2H_2O$ 40 41 e) Nitrogen (N) from feed or air is emitted as molecular 42 nitrogen 43 $N_2 \rightarrow N_2$ 44 45 Third, with these rules, the balanced chemical reaction for 46 combustion of a compound can be written.

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

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.

17 In the ATRS, the test sample is gasified and transported to a 18 high temperature reactor. In the high temperature reactor, 19 the sample vapors are subjected to controlled conditions for 20 residence time and temperature. As described in Sections 21 C.2.4.5 and C.2.4.6, combustion products will be monitored or 22 collected for quantitative analysis.

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

26

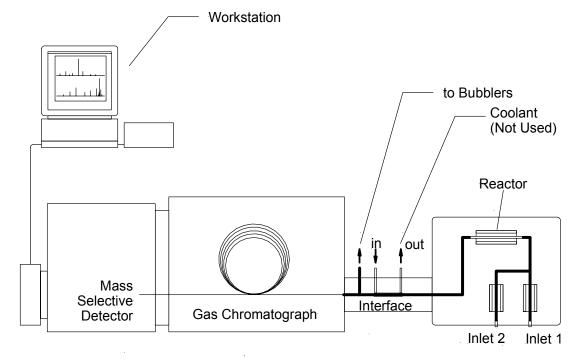
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16

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



28

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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 8 pyroprobe, a device designed to gasify samples by heating them at a fixed rate. The main gas flow will also be fed 9 via Inlet 1, and Inlet 2 will be used to feed supplemental 10 11 flow. 12 13 During combustion tests, the transfer line between the 14 pyroprobe and the reactor is heated and maintained above 15 200 $^{\circ}$ C. The reactor is housed within its own small tube 16 furnace and may be independently heated to as high as 1100 17 °C. (Actual conditions for this test program are presented 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 20 prevent cool regions where reactor products could otherwise 21 be lost through condensation. 22 23 The interface routes the combustion exhaust gas to the in-24 line gas chromatograph (GC) and mass selective detector 25 (MSD) or to sample collection for off-line analysis. For 26 combustion testing in this test program, the interface will 27 also be maintained above 200 °C. Exhaust gas monitoring for 28 this program is described in Section C.2.4.5. 29 30 C.2.4.3 Combustion Test Experimental Conditions 31 32 Each test substance composite will be subjected to 33 laboratory-scale incineration using the experimental 34 apparatus described in Section C.2.4.2. 35 36 C.2.4.3.1 Combustion Air 37 38 Synthetic air (mixture of 21% oxygen and 79% nitrogen) will 39 be used in place of compressed air to prevent potential 40 interference in the experimental system due to background 41 levels of CO_2 in compressed air. 42 43 C.2.4.3.2 Fuel 44 45 Methanol will be used, as needed, as a supplemental fuel to 46 ensure the presence of sufficient hydrogen to convert

1 fluorine to hydrogen fluoride (HF) and chlorine to hydrogen 2 chloride (HCl). 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 11 was known as wood alcohol. Therefore, methanol can be used in this experimental program as a surrogate for the paper 12 13 and wood fraction of MSW. 14 15 C.2.4.3.3 Operating Conditions 16 17 The target operating conditions for the high temperature 18 reactor during the combustion tests for each test substance 19 composite identified in Appendix A.3 are presented in Table 20 C.2-1. 21

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

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.

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.

34

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

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 5 temperature ramp-up) will be 750 °C or as needed to assure this section is 50 to 100 $^\circ C$ above the highest temperature 6 7 for complete gasification across the test substance composites as determined from the TGA results; see Section 8 C.2.3. This is necessary to assure complete gasification 9 of the sample of test substance composite and a common set 10 11 of experimental conditions across the test materials during combustion testing. 12 13 14 C.2.4.3.4 Blanks 15 16 A minimum of one thermal blank will be run prior to each 17 set of three combustion test runs for a given test 18 substance composite. Each thermal blank run will be at the corresponding combustion test conditions with all feeds 19 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 25 for each combustion test at key points during the test as 26 noted in the table. Each combustion test will be a minimum 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 31 TABLE C.2-2. COMBUSTION TEST MONITORING Parameter Key Time for Recording Before & after gasification Temperature-Reactor 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 Before & after gasification Gas flow rate-Inlet 2 Total Gas Flow rate Before & after combustion test Make-up Gas (He) Flow rate Before & after combustion test Pressure-Reactor Before & after gasification

32

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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1 The flow rate of the exhaust gas routed to the bubblers (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 6 by weighing the pyroprobe insert cartridge before and after 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 15 an oxygen monitor. CO_2 in exhaust gas will be monitored via 16 in-line GC, in-line MSD, or a continuous monitor; or 17 exhaust gas will be collected in Tedlar® bags for off-line 18 analysis of CO_2 . Carbon monoxide (CO) in exhaust gas will be monitored via in-line GC or a continuous monitor; or 19 20 exhaust gas will be collected in Tedlar® bags for off-line analysis of CO. Tedlar® bag samples may be collected at 21 22 the exit of the bubblers described in Section C.2.4.6. 23 24 C.2.4.6 Exhaust Gas Sampling 25 26 Gas samples for off-line analysis will be collected as 27 described in Appendix D.1, revised as necessary pursuant to 28 Appendix C.1.2.2 if applicable. 29 30 A minimum of 60 mL of bubbler aqueous solution composite is 31 expected from each combustion test. Of this, a minimum of 45 mL will be directed to PFOA analysis, and the remainder 32 33 will be directed to fluoride ion analysis. 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 40 from each combustion test collected as described in Section C.2.4.6 will be analyzed for fluoride ion via ion 41 42 chromatography using EPA Method 300.0. 43 44 C.2.4.7.2 PFOA 45 46 A portion of the composite bubbler aqueous solution sample from each combustion test collected as described in Section 47

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

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

be prepared following the outline in Appendix E.2 and will 1 be included in the test report. 2 3 4 C.2.5.6 Test Report Outline 5 The outline for the test report is presented in Appendix 6 7 E.3. All reporting discussed in Sections C.2.5.1 through C.2.5.5 will be included in this test report, as 8 applicable. 9

APPENDIX D.1 1 2 EXHAUST GAS SAMPLING VIA BUBBLERS 3 4 Gas samples for off-line analysis will be collected from a 5 vent line off the interface of the thermal reactor system described in Appendix C.2.4. Flexible (silicone or 6 7 equivalent) tubing will connect the vent line and a set of 8 bubblers. 9 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 15 total amount of solution at the beginning of each test run will be a minimum of 55 mL. The temperature of the gas 16 17 exiting the last bubbler will be monitored. 18 19 An additional bubbler (which is empty) will be added to the 20 front end of this series of bubblers to serve as a knock-21 out pot if calculations or preliminary measurements 22 indicate that greater than 10 mL of water will be produced 23 during the testing for a given material. 24 25 Upon completion of sample collection, the amount in each 26 bubbler will be weighed and recorded, and the contents of 27 the bubblers will be composited for subsequent analysis. 28 Additionally, the flexible tubing will be rinsed with 5 mL 29 of HPLC water to collect potential condensate in the tubing; this rinsate will be combined with the bubbler 30 composite prior to analysis. 31 32 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
    Spectrometry (LC/MS/MS) in accordance with "Method of
 8
 9
    Analysis for the Determination of Ammonium
    Perfluorooctanoate (APFO) in Water Revision 1" (Exygen
10
11
    method) revised per the section-by-section comments listed
    in Section D.2.4 below. These revisions are necessary to
12
13
    adapt a method originally developed for liter quantity
14
    water samples to samples related to testing described in
15
    Appendix C.
16
17
    The testing programs described in Appendix C are expected
18
    to generate samples of aqueous solution, methanol (e.g., as
    used for extraction or rinsing), and corresponding blanks.
19
20
    The expected sample size for aqueous solution samples (from
    exhaust gas bubbler sample collection) available for
21
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
28
    cartridge with methanol. Quantification of PFOA is
29
    accomplished by electrospray liquid chromatography/tandem
30
    mass spectrometry (LC/MS/MS) analysis.
31
    D.2.3 Reporting
32
33
34
    The target limit of quantitation (LOQ) for this study with
35
    this method is 50 ng/L based on prior work with water
    samples where an 8-fold concentration via extraction using
36
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
40
    performed, the target LOQ for this study is 400 ng/L.
41
42
    Sections 4.5.4 and 5 of the Exygen method explain reporting
    for field samples such as bubbler aqueous solution
43
44
    composites, which are distinct from blanks and spikes, as
45
    follows:
46
47
         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).

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

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

20

15

7

21 22 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 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, the methol to extraction using the C₁₈ SPE cartridge described in section 4.4 of the sample is not amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of the sample is not amenable to extraction using the C₁₈ SPE cartridge described in section 4.4 of 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. |

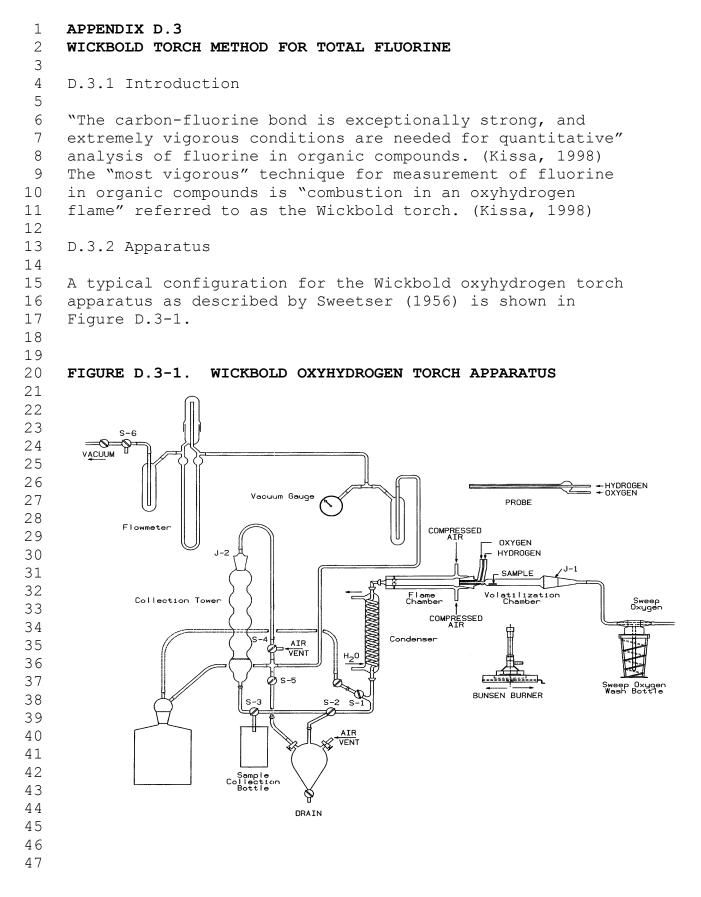
| 3.3 | • The following text will be used in place of |
|----------------|---|
| Notes, | Note 1 with respect to the PFOA analysis |
| Note 1 | conducted for this testing program: |
| | In order to avoid contamination, the use of disposable labware (tubes, pipets, etc.) is required. |
| 3.3 | • The following text will be used in place of |
| Notes, | Note 4 with respect to the PFOA analysis |
| Note 4 | 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 | • Where the available amount of sample is |
| opening | expected to be much less than 1 liter, |
| text | insufficient sample is available to prepare |
| prior to | the fortified matrix spikes described in the |
| 3.5.1 | 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, | • Where the available amount of sample is |
| item b | 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 |
| Λ 2 | 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 |

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

1 2 3

2 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
January 2003. (EPA Docket ID OPPT-2003-0012-0040)



1 D.3.3 Method Description 2 3 The sample size for the standard sample boat is up to 20 mg 4 for a solid or up to 5 mL for a liquid. 5 6 With the oxyhydrogen torch in operation, the sample is 7 pyrolyzed or vaporized with a Bunsen burner moving on a rail below the volatilization chamber. The vapors and 8 pyrolysis products are swept through the oxygen-hydrogen 9 flame chamber operating at up to approximately 2000 °C to 10 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 the Wickbold Torch method is 0.5 ppm (0.5 mg/kg). The 19 20 accuracy of this method for determination of total fluorine in fluorinated polymers is exemplified by total fluorine 21 values of 75.35% to 75.84% for PTFE with known total 22 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 31 safety devices by well-trained personnel at a qualified 32 laboratory. 33 34 D.3.5 References 35 36 Kissa, E. "Analysis of Anionic Fluorinated Surfactants", 37 Chapter 8 in Anionic Surfactants: Analytical Chemistry -38 2nd Edition, Revised and Expanded, edited by John Cross. Marcel Dekker Surfactant Science Series, volume 73, 1998. 39 40 41 Sweetser, P. B. "Decomposition of Organic Fluorine 42 Compounds by Wickbold Oxyhydrogen Flame Combustion Method", 43 Analytical Chemistry, vol. 28, pp. 1766-1768, 1956.

1 APENDIX D.4

2 WASTE INCINERATION AND OPERATION CONDITIONS

3

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.

8 9

D.4.1 Types of Incinerators

10

11 D.4.2.1 Municipal Waste Combustors

12

13 According to the Integrated Waste Services Association 14 (IWSA), there are a total of 98 waste-to-energy facilities 15 operating municipal waste combustors (MWCs) in the U.S. as 16 of 2002. (IWSA 2002) Table D.4-1 summarizes the number and 17 annual capacity of these units by type of technology 18 employed.

19

20 Table D.4-1. MWCs in 2002

| 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% | |
| Total | 98 | 29.4 | 100.0% | |

21

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

32

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

1 have reciprocating grates or roller grates to move the waste through the combustion chamber. (EPA 1996a) 2 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 temperature of 2238 $^{\circ}\text{F}$ (1226 $^{\circ}\text{C})$ over a 2 second. The 8 9 corresponding gas temeperature at the 2 second level from this figure is 1750 $^{\circ}$ F (954 $^{\circ}$ C). 10 11 12 A report on the Warren County, New Jersey mass burn water 13 wall MWC indicates that the design gas temperature between 14 the grate and secondary air inject was greater than 2000 $^{\circ}F$ (1093 $^{\circ}$ C) over a gas residence time of an additional 2.2 15 16 seconds. (Schuetzenduebel and Nobles 1990) This report 17 also shows that this MWC was designed for 2 seconds 18 residence time above 1800 °F (982 °C) between the introduction of secondary air and the exit of the furnace 19 20 section. (Schuetzenduebel and Nobles 1990) The temperature 21 profile (Figure 21) in the temperature correlation test report (Schutzenduebel 1989) for this MWC shows the full 22 23 load gas temperature at the secondary air injection point 24 is 2650 $^{\circ}$ F, and the gas temperature at the 2-second point is 25 1850 °F. Therefore, testing indicates an average temperature of 2250 $^{\circ}$ F (1232 $^{\circ}$ C) over this 2 second gas 26 27 residence time for the Warren County unit. A related 28 report for the Warren County MWC by the design firm 29 indicates that the exhaust gas oxygen concentration is 30 nominally 10% (dry basis). (Blount Energy Resource Corp. 31 1989) 32 Information from these 2 MWCs demonstrates that the average 33 34 gas temperature across a 2 second residence time for mass 35 burn MWCs is conservatively expected to be greater than 1100 °C. 36 37 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% moisture (water). (Clean Air Engineering, 1997) 41 42 43 As indicated in Table D.4.1, mass burn units account for over 76% of the municipal solid waste incinerated in the 44 45 U.S.

1 2 3

D.4.2.1.2 RDF MWC

Furnace temperatures as well as flue gas oxygen and moisture
(H₂O) 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.

9

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

| ΤU | TADIE D.4-2. RDF | MMC | - MIG | -conne | CLICUL | | | | |
|----|---|----------|-------|-------------------|-------------------|---------|--------------|---------|-------|
| | 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 | 21 10 | | | 11 00 | 22 00 | 22 00 | | |
| | 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 |
| 11 | | | | | | | | | |
| 12 | The average oper | ating | cond | itions | for th | nis RDF | unit a | cross | |
| 13 | the range of ste | am lo | ads a | re 101 | 6 °C, 8 | .4% 02 | (drv ba | asis), | |
| 14 | and 14.1% moistu | | | | , - | 2 | (- <u>1</u> | / / | |
| 15 | | | | | | | | | |
| 16 | Examination of t | ho ro | nort | and MW | C tompo | naturo | monito | ring | |
| 17 | practices indica | | - | | - | | | - | - |
| | - | | | | - | | | - | - |
| 18 | combustion zone | | - | | | | | | |
| 19 | determine the av | - | | | | | - | | |
| 20 | across a 2 secon | - | | | | | | ry to | |
| 21 | understand the t | ime-t | emper | ature | profile | e of th | e MWC. | | |
| 22 | | | | | | | | | |
| 23 | Since waste combustion in this and most other RDF units in | | | | | | | | |
| 24 | the U.S. involves burning on the grate (EPA 1996a) similar | | | | | | | | |
| 25 | to the operation | of m | ass b | urn MW | Cs, the | e time- | tempera | ature | |
| 26 | profile in an RDF unit is expected to be similar to that | | | | | | | | |
| 27 | described in Sec | tion | D.4.2 | .1.1 a | bove. | Based | on this | 3 | |
| 28 | similarity and t | he te | mpera | tures | in Tabl | Le D.4- | 2, the | average | e |
| 29 | gas temperature | acros | sa2 | secon | d resid | dence t | ime for | RDF | |
| 30 | units is conservatively expected to be greater than 1100 °C. | | | | | | | | |
| 31 | units is conscivatively expected to be greater than 1100 c. | | | | | | | | |
| 32 | As indicated in | Table | D.4. | 1. RDF | units | accoun | t for | | |
| 33 | As indicated in Table D.4.1, RDF units account for approximately 22% of the municipal solid waste incinerated | | | | | | | | |
| 34 | in the U.S. | O OL | | unitorp | ar sori | La Wabe | | leracea | |
| 35 | | | | | | | | | |
| 36 | D.4.2.1.1 Modula | TO MINTO | | | | | | | |
| | D.4.2.1.1 Modula | LE MWC | | | | | | | |
| 37 | | | | | | | | | |
| 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 | | | | | | | | |

1 secondary chamber. (EPA 1996a) EPA notes that the 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 $^{\circ}$ F (982 $^{\circ}$ C) to 2000 $^{\circ}$ F (1093 $^{\circ}$ C), flue gas oxygen 8 9 concentrations in the range of 10% to 13% (dry basis), and flue gas moisture in the range of 10% to 15% (Pace 10 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 15 secondary chamber average gas temperatures for modular MWCs 16 are expected to be 1000 $^{\circ}C$ or greater. 17 18 As indicated in section D.4.1, such modular units are generally small MWCs and account for less than a total of 19 20 2% of the municipal solid waste incinerated in the U.S. 21 22 D.4.2.1.4 MWC Summary 23 24 Considering the relative quantities of municipal waste 25 burned annually in each type of MWC and the data in this 26 section, typical operating conditions for the high 27 temperature zone of most MWCs are >1000 °C average temperature across 2 second residence time with exit gas 28 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 34 controlled air medical waste incinerators has been reported 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 σf medical waste incinerators to sustain temperatures in 38 the range of 985 to 1095 $^\circ \rm C$ and that combustion air at 150 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 notes that the average moisture content in HMIWI flue gas 43 44 was about 10 % based on available data, and EPA states "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

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

43

44 Over 99% of large MWC capacity operates with a spray dryer 45 absorber/scrubber. (IWSA 2003) Approximately 80% of large 46 MWC capacity operates using carbon injection as part of the 47 pollution control system. (IWSA 2003) Due to requirements

INTERESTED PARTY REVIEW FINAL DRAFT 02-27-04 in the NSPS (EPA 2000b) and EG (EPA 200c) for small MWCs, 1 small MWCs planning continued operation are generally 2 upgrading or have upgraded their pollution control 3 4 equipment to add spray dryer absorbers or other acid gas 5 control and carbon injection. 6 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. 10 11 D.4.4 Summary 12 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 22 are as follows: 23 >1000 °C Average Temperature Residence Time >2 sec O₂ concentration in exhaust gas 10% (dry basis) H₂O concentration in exhaust gas 15%

24

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

1 References

2 Blount Energy Resource Corp. Correlation Procedure for 3 4 Continuously Monitoring Furnace Temperatures (Warren County 5 Resource Recovery Facility), March 22, 1989. 6 7 Clean Air Engineering. Test Report for Covanta of Fairfax, 8 Inc. I-95 Energy/Resource Recovery Facility, 1997. 9 Donnelly, J.R. Waste Incineration Sources: Municipal Waste 10 11 Combustion. In: W.T., ed., Air Pollution Engineering 12 Manual, 2nd edition. Air and Waste Management Association. 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 20 Combustion Assessment: Technical Basis for Good Combustion 21 Practice, EPA 600/8-89-063, August 1989. 22 23 EPA. Medical Waste Incinerators - Background Information 24 for Proposed Standards and Guidelines: Control Technology 25 Performance Report for New and Existing Facilities, EPA-26 453/R-94-044a, July 1994. 27 28 EPA. Medical Waste Incinerators - Background Information 29 for Proposed Standards and Guidelines: Model Plant 30 Description and Cost Report for New and Existing 31 Facilities, EPA-453/R-94-045a, July 1994. 32 33 EPA. Decision Maker's Guide to Solid Waste Management, Volume II, Chapter 8, 1995. 34 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 40 EPA. Medical Waste Incineration Emission Test Report: Weeks Memorial Hospital, Lancaster, New Hampshire, EMC 41 42 Report 96-MWI-11, March 1996. 43 44 EPA. Standards of Performance for New Stationary Sources 45 and Emission Guidelines for Existing Sources: 46 Hospital/Medical/Infectious Waste Incinerators, 62 Federal 47 Register 48346, September 15, 1997.

1

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APPENDIX E.1 (Fluorotelomers)
 1
    OUTLINE FOR INTERIM PROGRESS REPORTING
 2
 3
 4
 5
    Title:
              Enforceable Consent Agreement for the Laboratory-
              Scale Incineration Testing of Fluorotelomer Based
 6
 7
              Polymers - Interim Report
 8
 9
    OPPT Docket ID No: OPPT-2004-0001
10
11
    Date of Interim Report: [ date ]
12
13
14
15
    This Report covers the period from [date] to [date]
16
17
18
         List or description of significant ECA Test Program
    1)
         milestones during this period:
19
20
21
22
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24
25
    2)
        Description of Difficulties: (If none indicate N/A)
26
27
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31
32
33
    3)
         Actions taken in response to difficulties: If none
34
         indicate N/A)
35
36
37
38
39
40
         Other information relevant to the progress of the
41
    4)
42
         testing program: (If none indicate N/A)
43
```

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

2 OUTLINE FOR RELEASE ASSESSMENT REPORT

3

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

- 20 21
- 1.0 Introduction
- 22
- 23 24

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- 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.
- 40 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

| 1 | | |
|----|-----|---|
| 1 | | operational parameters. Cross-reference to or |
| 2 | | submission of relevant parts of Appendix D.4 of this |
| 3 | | ECA can satisfy this provision. |
| 4 | | |
| 5 | | • Description of the post-combustion air pollution |
| | | |
| 6 | | control equipment (e.g., lime scrubbing, carbon |
| 7 | | adsorption) employed by typical operating full-scale |
| 8 | | waste incineration process(es) as applicable. |
| 9 | | |
| 10 | 4.0 | Extrapolation of laboratory test results to the |
| 11 | | typical waste incineration process(es), as applicable, |
| 12 | | described in Section 3.0 (above) for each test |
| 13 | | |
| | | substance composite to be evaluated. |
| 14 | | |
| 15 | | The relevance of the subject test substance |
| 16 | | composite to MWCs and/or medical waste incinerators. |
| 17 | | |
| 18 | | · The estimated concentration of the subject test |
| | | • The estimated concentration of the subject test |
| 19 | | substance composite to the applicable type(s) of |
| 20 | | waste incinerator. Available information on |
| 21 | | hydrogen fluoride concentration in waste incinerator |
| 22 | | exhaust can provide the basis for an upper bound on |
| 23 | | this estimated concentration. |
| 24 | | |
| | | |
| 25 | | A description of the extrapolation. |
| 26 | | |
| 27 | | A description of any assumptions used. |
| 28 | | |
| 29 | | • Any unique quelitative en questitative descriptors |
| | | • Any unique qualitative or quantitative descriptors |
| 30 | | of the test, the testing equipment, and the results |
| 31 | | deemed necessary for informative review of the test |
| 32 | | and test results. |
| 33 | | |
| 34 | 5.0 | Sensitivity Analysis |
| 35 | | |
| | | · Accordment of the impact of menichility |
| 36 | | • Assessment of the impact of variability |
| 37 | | (quantitative) and uncertainty (qualitative) in each |
| 38 | | parameter on the evaluation results. |
| 39 | | |
| 40 | 6.0 | Conclusions |
| 41 | | |
| 42 | 7.0 | References |

42 7.0 References

| 1 | |
|----------|--|
| 2 | OUTLINE OF TEST REPORT ^{*,**} |
| 3 4 | |
| 4 5 | Table of Contents |
| 6 | Table of concents |
| 7 | Executive Summary |
| 8 | |
| 9 | Introduction |
| 10 | |
| 11 | I. Phase I PFOA Transport Testing |
| 12 | |
| 13 | 1. Experimental Apparatus |
| 14 | 2. Description of Test Conditions (including deviations |
| 15 | from protocol) |
| 16 | 3. Documentation of PFOA Standard |
| 17 18 | 4. Analytical Results 4.1 PFOA |
| 19 | 4.1 FFOA 4.2 Total Fluorine |
| 20 | 5. Transport Efficiency |
| 21 | 5.1 PFOA |
| 22 | 5.2 Total Fluorine |
| 23 | 6. Discussion of Results |
| 24 | 7. Conclusions |
| 25 | |
| 26 | II. Phase II Incineration Testing (provided Phase II is performed) |
| 27 | |
| 28 | 1. Documentation of Test Substance Composites |
| 29 | 2. Elemental Analysis Results |
| 30 | 3. Combustion Stoichiometry Results |
| 31 | 4. TGA Results |
| 32 33 | 5. Combustion Testing 5.1 Experimental Apparatus |
| 34 | 5.2 Description of Test Conditions (including deviations |
| 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) |

| 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 |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | * | Test Report will include this information (as applicable) |
| 12 | | but not necessarily in this order. |
| 13 | | |
| 14 | ** | References to literature in this report will include full |
| 15 | | citations. |
| | | |

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[Note to the Interested Parties: As described in the transmittal note, the Interested Parties have been asked to specifically comment on this point. The following describes the remaining issue regarding Appendix F]

DESCRIPTION OF INCINERATION ECAS QAPP ISSUE

7 The Incineration ECA Drafting Committee(s) have been unable to reach 8 consensus on Appendix F. Appendix F is an effort supported by the Companies 9 to list the required elements of the QAPP(s) and to address how these will be 10 addressed in the QAPP. In discussions to date, EPA and the companies have 11 reached a common understanding on several points. The QAPP(s) will be 12 prepared in accordance with EPA document EPA QA/R5: EPA Requirements for 13 Quality Assurance Project Plans ("QA/R5"). The QAPP(s), as described under 14 Appendix F, would address the elements in QA/R5 in the order in which they 15 appear in the EPA QA/R5 guidance document. The QAPP(s) and Appendix F would 16 cover all activities under this ECA, including creation of the composite test 17 substances that will be assembled at designated facility(ies). Finally, EPA 18 and the companies agree that it should not be necessary to repeat in a QAPP 19 information that appears elsewhere. EPA and the companies agree that QAPP 20 elements may be satisfied by cross-referencing to applicable portions of the 21 ECA agreement including any of the appendices as appropriate or to the GLP 22 regulations at 40 CFR 792. While both groups agree that cross-referencing is 23 appropriate where the QAPP elements overlap with the ECA, ECA appendices or 24 GLP requirements, the level of specificity for cross-referencing that would 25 be included in Appendix F is still under discussion. 26

EPA and the Companies have been unable to agree on the scope of Appendix F. The companies believe Appendix F should be a definitive outline of the required contents of the QAPPs. In other words, something that is not required by Appendix F is not required in a QAPP. The companies believe that having to address items not specified in Appendix F would be equivalent to a renegotiation of the testing protocols that the Drafting Committee has complied as appendices to the ECA.

35 While EPA and the Companies have been working since November to develop 36 Appendix F, EPA believes that it is not feasible to develop anything more 37 than an outline of what the QAPP could include and that this has already been 38 accomplished. Up-front investment in developing study protocols and detailed 39 procedures are a necessary part of the ECA development process to clarify the 40 scope and intent of the testing program. Nevertheless, EPA believes this up-41 front effort should not be viewed as a means by which pre-approval of a 42 QAPP(s) could be established. How Test Sponsors address data quality is 43 unique to the characteristics of each laboratory or facility participating in 44 the testing program, the equipment available at each facility, the nature of 45 the testing, and the systems that the Test Sponsor establishes to collect, 46 communicate, and report data across individual elements of the testing 47 program. EPA believes that Appendix F could be acceptable as an outline for 48 the QAPP(s) if the right hand column is deleted from the table in Appendix F; 49 or, if the heading for the right hand column were changed from: "Required 50 Content of QAPP(s) for the ECA Incineration Testing" to read: "Minimum 51 Submission Requirements for QAPP(s)." A similar change would also be needed 52 to the title of the appendix. 53

54 EPA and the Companies are specifically seeking input from interested 55 parties on the appropriate scope of Appendix F. EPA and the Companies also 56 welcome alternative approaches that the interested parties may have regarding 57 how to improve Appendix F.

1 APPENDIX F

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

- 3 REQUIRED CONTENT
- 4

| 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/ | to be satisfied by cross- |
| Background | 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 | to be satisfied by cross- |
| Criteria | 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 | see element A5 |
| (Experimental Design) | |
| 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 | to be included in QAPP |
| Custody | consistent with Appendix A |
| B4 Analytical Methods | to be satisfied by cross- |

| | 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 |
| B6 Instrument/Equipment | to be included in QAPP in |
| Testing, Inspection, and | summary form for chemical |
| Maintenance | analysis equipment for the |
| | analytical methods for |
| | element B4 above |
| D7 Instrument /Equipment | to be included in QAPP in |
| B7 Instrument/Equipment | ~ |
| 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 |
| | 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 |
| bro baca management | - |
| | 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 |
| | Sacrorrea by comprisined |

| C2 Reports to Management | <pre>with applicable GLP requirements; for other facilities, to be included in QAPP 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</pre> |
|---|--|
| D: DATA VALIDATION AND USABILITY | ~ |
| D1 Data Review, Verification, and Validation | 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 |
| D2 Verification and Validation Methods | 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 consistent with Appendices A, C.1, C.2, D.2, D.3 as applicable |
| D3 Reconciliation with User Requirements | to be satisfied by cross- reference to Appendices C.2.5.5 and E.2, as applicable |

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| 1 | APPENDIX G (Fluorotelomers) | |
|----------|-----------------------------|--------------------------|
| 2 | COPY OF EPA ORDER | |
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| 5 | UNITED STATES | |
| 6 | | ON AGENCY |
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| 31 | | . Johnson, |
| 32 | 1 | Administrator |
| 33 | | ntion, Pesticides, |
| 34 | | Substances |
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| 1 | APPENDIX G (Fluoropolymers) |
|----------|--|
| 2 | COPY OF EPA ORDER |
| 3 | |
| 4 5 | UNITED STATES |
| 6 | ENVIRONMENTAL PROTECTION AGENCY |
| 7 | ENVIRONMENTAL PROTECTION AGENCI |
| 8 | TESTING CONSENT ORDER FOR THE LABORATORY-SCALE INCINERATION |
| 9 | TESTING OF FLUOROPOLYMERS |
| 10 | |
| 11 | Docket No. OPPT-2003-0071 |
| 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 17 | Environmental Protection Agency (EPA) issues this testing 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 | fluoropolymer test substance composites listed in Appendix A of |
| 22 | the ECA. |
| 23 | |
| 24 | |
| 25 | |
| 26 | |
| 27 | |
| 28 29 | |
| 30 | |
| 31 | Date Stephen L. Johnson, |
| 32 | Assistant Administrator |
| 33 | For Prevention, Pesticides, |
| 34 | And Toxic Substances |