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Subject: revised Appendices E.2 and C.2 based on Jan. 21 FP incin drafting
comm call

Colleagues,

As requested, I am sending the revised versions of Appendices E.2 and C.2 based on edits discussed during the fluoropolymer incineration testing drafting committee call on the morning of January 21 (EST) without tracking changes.

The opening sentence of E.2 now references Part VII.B of the ECA and Section 2 of E.2 has been revised to replace "test condition" with "applicable test substance composite" for clarification.

(See attached file: App E.2 release assess outline draft 1-21-04.pdf)

The end of Section C.2.5.5 of Appendix C.2 has been revised to clarify that if triggered the Release Assessment report will be included in the test report. Also, in order to clarify the meaning of "test report" in Section C.2.5, I have defined it as final report for incineration testing at first mention in C.2.5 at C.2.5.1.

(See attached file: App C.2 incin testing draft 1-21-04.pdf)

Thank you again for your comments.

If you have any questions, please let me know.

Best Regards,

Robert Giraud

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1 APPENDIX C.2**2 INCINERATION TESTING****4 C.2.1 Elemental Analysis****6 C.2.1.1 Introduction**

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.

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

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

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 provide estimated values.

36 C.2.1.2 Total Fluorine

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

41 Based on manufacturing process knowledge, the levels of
42 total fluorine in the components of test substance
43 composites are orders of magnitude higher than the
44 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 will be considered to be the same as the total fluorine
48 value.

C.2-1

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

4 C.2.1.3 Carbon and Hydrogen

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

14 C.2.1.3.1 Theoretical Determination

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

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

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

27 C.2.1.3.2 Empirical Determination

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

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

C.2-2

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1 The carbon content of the test substance composite can be
2 measured by determining the carbon dioxide (CO₂) generated
3 by the oxidation of the sample. This oxidation may be
4 accomplished by high temperature combustion, catalytic
5 combustion, or wet chemical oxidation. The CO₂ is measured
6 directly by an infrared detector or a thermal conductivity
7 detector, via absorption into a suitable solution (e.g.,
8 potassium hydroxide) and gravimetric determination, or by
9 conversion to methane for measurement via a flame
10 ionization detector.

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

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

33 34 C.2.1.4 Moisture

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

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

C.2-3

1 The moisture value is calculated as the loss in weight
2 (difference between the starting weight of sample and the
3 final weight of sample) divided by the starting weight of
4 sample. Similarly, a solids value can be calculated as the
5 final weight of sample divided by the starting weight of
6 sample.

7

8 **C.2.2 Combustion Stoichiometry**

9

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

13

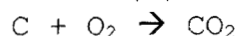
14 First, the weight percent values from Section C.2.1 are
15 converted to molar quantities on a dry basis.

16

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

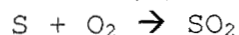
22

23 a) All carbon (C) in feed converts to carbon dioxide (CO₂)



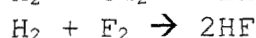
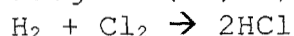
25

26 b) All sulfur (S) in feed converts to sulfur dioxide (SO₂)



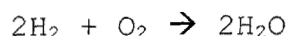
28

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



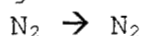
32

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



37

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

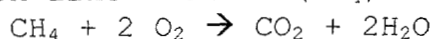


41

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

44

45 For example, the resulting reaction equation for a
46 hydrocarbon like methane (CH₄) is



C.2-4

5

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

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

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

21 22 **C.2.3 Thermogravimetric Analysis**

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

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

38 39 **C.2.4 Combustion Testing**

40 41 **C.2.4.1 Test Objective**

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

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

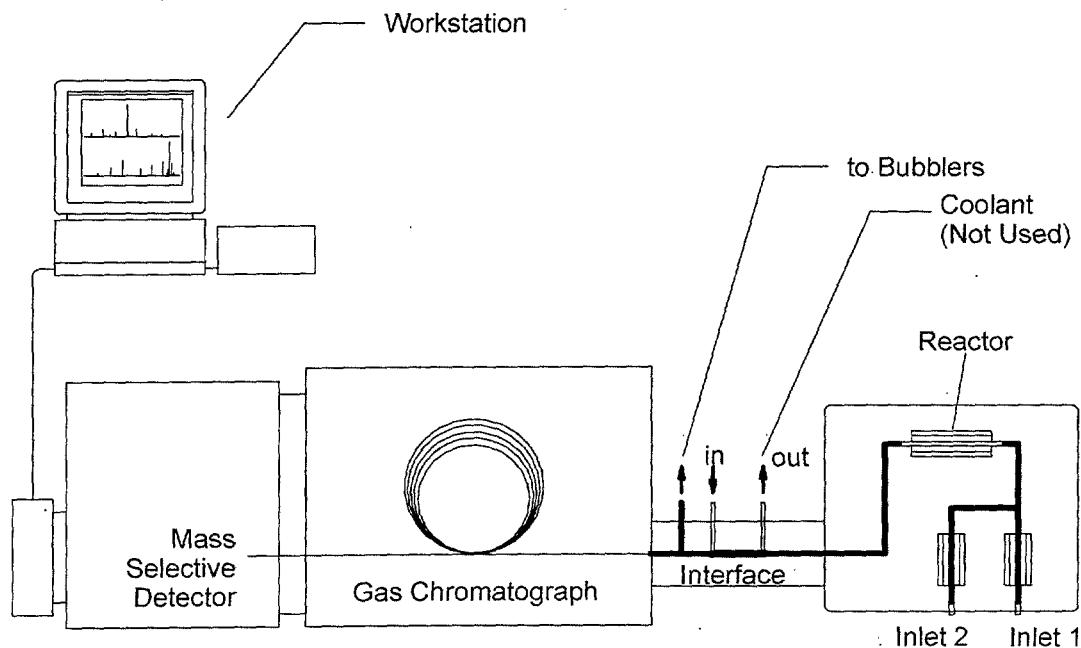
3 4 C.2.4.2 Experimental Apparatus

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

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

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

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



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

C.2-6

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

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

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

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

27
28 C.2.4.3 Combustion Test Experimental Conditions

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

33
34 C.2.4.3.1 Combustion Air

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

40
41 C.2.4.3.2 Fuel

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

47

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

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

11 12 C.2.4.3.3 Operating Conditions

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

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

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

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

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

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

42

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

10 C.2.4.3.4 Blanks

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

17 C.2.4.4 Process Monitoring

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

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

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

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

29 The flow rate of the exhaust gas routed to the bubblers (see
30 Section C.2.4.5.2) will be determined based on the flow
31 measurements listed in Table C.2-2.
32

1

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

5

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

7

8 C.2.4.5 Exhaust Gas Monitoring

9

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

20

21 C.2.4.6 Exhaust Gas Sampling

22

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

25

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

30

31 C.2.4.7 Exhaust Gas Analysis

32

33 C.2.4.7.1 Fluoride Ion

34

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

39

40 C.2.4.7.2 PFOA

41

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

46

47 As described in Appendix D.2, composite bubbler aqueous

C.2-10

1 solution sample results less than method detection limit
2 (MDL) will be reported as not detected (ND), results
3 between MDL and the limit of quantitation (LOQ) will be
4 reported as not quantifiable (NQ), and numerical values
5 will not be reported.

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

13
14

15 C.2.5 Reporting of Results

16

17 C.2.5.1 Elemental Analysis Results

18

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

23

24 C.2.5.2 Combustion Stoichiometry Results

25

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

29

30 C.2.5.3 TGA Results

31

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

36

37 C.2.5.4 Combustion Test Results

38

39 C.2.5.4.1 Process Monitoring

40

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

44

45 C.2.5.4.2 Exhaust Gas Monitoring

46

47 Exhaust gas O₂, CO and CO₂ monitoring results will be

C.2-11

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

5

6 C.2.5.4.3 Exhaust Gas Analytical Results

7

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

10

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

18

19 C.2.5.4.3.1 Fluoride

20

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

25

26 C.2.5.4.3.2 PFOA

27

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

31

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

36

37 C.2.5.5 Release Assessment

38

39 In the event that PFOA is reported for the exhaust gas
40 bubbler aqueous solution at a concentration above the LOQ
41 for the three runs for a given test substance composite, a
42 release assessment report for the subject material will be
43 prepared following the outline in Appendix E.2 and will be
44 included in the test report.

45

46

C.2-12

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APPENDIX E.2**OUTLINE FOR RELEASE ASSESSMENT REPORT**

As described in Part VII.B, footnote 3 and Appendix C.2.5.5 of this ECA, if PFOA is reported for the exhaust gas bubbler aqueous solution at a concentration above the LOQ for the three runs for a given test substance composite, then the potential for release from full-scale municipal and/or medical waste incineration, as applicable, (including application of air pollution controls) of the subject material in the United States will be assessed to put the data into perspective. At a minimum, the report will follow the general outline described below and will state assumptions, document the basis for the assumptions made, quantitatively estimate the variability of calculated estimates (based on the variability of the parameters in the evaluation), and qualitatively discuss the uncertainty of calculated estimates.

1.0 Introduction

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

2.0 Summary of study results

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

3.0 Discussion

- Description of the combustion section of the applicable waste incineration process(es) being evaluated (MWC and/or medical waste incinerator) including the rationale for selecting test target temperature(s) and description of typical operational parameters. Cross-reference to or

E.2-1

1 submission of relevant parts of Appendix D.4 of this
2 ECA can satisfy this provision.

3

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

8

9 **4.0 Extrapolation of laboratory test results to the**
10 **typical waste incineration process(es), as applicable,**
11 **described in Section 3.0 (above) for each test**
12 **substance composite to be evaluated.**

13

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

16

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

23

- 24 • A description of the extrapolation.

25

- 26 • A description of any assumptions used.

27

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

32

33 **5.0 Sensitivity Analysis**

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

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39 **6.0 Conclusions**

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41 **7.0 References**

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