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cc:

Subject: Re: materials and call-in number for fluoropolymers incin testing ECA confcall on Wed Jan 21 -- App C.1

Colleagues,

With apologies, this note conveys the revised draft Appendix C.1 with strike and insert as agreed to during the Jan. 13 drafting committee call that was intended to be included near the end of yesterday's e-mail message.

(See attached file: App C.1 transport test draft 1-19-04.pdf)

Best Regards,

Robert Giraud

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App C.1 transport test draft 1-19-04.

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1 APPENDIX C.1

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2 **PFOA TRANSPORT TESTING**

4 C.1.1 Significance

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

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

16 C.1.2 Experimental Plan

18 C.1.2.1 Base Plan

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

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

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

36 The sample size of the PFOA standard to be gasified will be 37 less than 5 mg. The reactor exhaust gas will be collected into bubbler aqueous solution as described in Appendix D.1 38 (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 42 be analyzed for PFOA as described in Appendix D.2. In 43 order to provide a second way of demonstrating quantitative 44 transport, this aqueous solution composite will also be 45 analyzed for total fluorine as described in Appendix D.3. 46 (Testing for total fluorine is included due to possibility 47 of thermal degradation of PFOA under transport test

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1 conditions.) Therefore, for this transport testing the 2 amount of PFOA fed to the thermal reactor system will be 3 sufficiently high to assure that the total fluorine input to the thermal reactor system will be greater than 140% of 4 the mass corresponding to the limit of quantitation (LOO) 5 6 for total fluorine in the aqueous solution composite. (The 7 LOQ for total fluorine in aqueous solution is much higher 8 than the LOQ for PFOA in aqueous solution.) 9 10 The amount of PFOA and total fluorine in the thermal reactor system exhaust gas will be determined via analysis 11 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 % PFOA TE = mass of PFOA in aqueous solution composite * 100 25 (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 C.1.2.2 Contingent Testing 34 35 If the transport efficiencies for both PFOA (equation 1) 36 37 and total fluorine (equation (2)) are less than or equal to 70%, then additional work will be performed. 38 This 39 additional work will be performedin a step-wise fashion to determine if consideration of one or more of the following 40 41 procedural revisions enables achievement of 70% transport efficiency as follows: 42 43 The flexible tubing between the thermal reactor 44 Step 1. system and the bubbler assembly from the experiment 45 described in Section C.1.2.1 would be 46 quantitatively rinsed with methanol. 47 This methanol rinsate would be analyzed for PFOA (as described in 48

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Appendix D.2) and/or for total fluorine (as 1 2 described in Appendix D.3). Revised transport 3 efficiency (TE) as a percentage for PFOA (equation 3) and/or total fluorine (equation 4) would be · 4 5 computed by including the mass of analyte in the 6 methanol rinse in the numerator as follows: 7 8 mass_{PFOA} out 9 % PFOA TE = ----- * 100 (3)10 mass_{PFOA} in 11 12 where $mass_{PFOA}$ out = mass of PFOA in bubbler 13 aqueous solution composite 14 + mass of PFOA in methanol 15 rinse 16 17 and mass_{PFOA} in = mass of PFOA fed to thermal 18 reactor system 19 20 masstotal F out % Total F TE = ----- * 100 21 (4)22 mass_{total F} in 23 where $mass_{total F}$ out = mass of total F in 24 25 bubbler aqueous 26 solution composite 27 + mass of total F in 28 methanol rinse 29 30 calculated mass of and mass_{total F} in = 31 total F in PFOA fed to 32 thermal reactor system 33 34 Step 2 (if necessary) The experiment described in Section 35 C.1.2.1 would be repeated with reagent(s) (e.g. NaOH) added to the 36 bubbler aqueous solution to determine 37 if reagent addition enhances analyte 38 absorption, thereby improving transport 39 efficiency. Transport efficiency would 40 be calculated using equation (1) and/or 41 (2) above. The impact of reagent 42 addition on LOQ for PFOA analysis 43 described in Appendix D.2 would be 44 determined. 45 46 47 C.1.3 Reporting of Results 48 Following completion of PFOA transport testing as described 49 in this appendix and prior to beginning incineration 50 51 testing described in Appendix C.2, a letter report will be C.1-3

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

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

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