5PSS

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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

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

APPENDIX C.1
PFOA TRANSPORT TESTING

C.1.1 Significance

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

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

C.1.2 Experimental Plan

C.1.2.1 Base Plan

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

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

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

The sample size of the PFOA standard to be gasified will be less than 5 mg. The reactor exhaust gas will be collected into bubbler aqueous solution as described in Appendix D.1 (including an HPLC water rinse of the flexible tubing [used to connect the thermal reactor system and the bubbler assembly] into the aqueous solution composite), which will be analyzed for PFOA as described in Appendix D.2. In order to provide a second way of demonstrating quantitative transport, this aqueous solution composite will also be analyzed for total fluorine as described in Appendix D.3. (Testing for total fluorine is included due to possibility of thermal degradation of PFOA under transport test

conditions.) Therefore, for this transport testing the amount of PFOA fed to the thermal reactor system will be sufficiently high to assure that the total fluorine input to the thermal reactor system will be greater than 140% of the mass corresponding to the limit of quantitation (LOQ) for total fluorine in the aqueous solution composite. (The LOQ for total fluorine in aqueous solution is much higher than the LOQ for PFOA in aqueous solution.)

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

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

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

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

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

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

C.1.2.2 Contingent Testing

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

Step 1. The flexible tubing between the thermal reactor system and the bubbler assembly from the experiment described in Section C.1.2.1 would be quantitatively rinsed with methanol. This methanol rinsate would be analyzed for PFOA (as described in

Appendix D.2) and/or for total fluorine (as described in Appendix D.3). Revised transport efficiency (TE) as a percentage for PFOA (equation 3) and/or total fluorine (equation 4) would be computed by including the mass of analyte in the methanol rinse in the numerator as follows:

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

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

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

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

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

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

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

C.1.3 Reporting of Results

Following completion of PFOA transport testing as described in this appendix and prior to beginning incineration testing described in Appendix C.2, a letter report will be

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

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