TECHNICAL MEMORANDUM

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DATE: February 13, 2002

SUBJECT: $PM_{2.5}$ Quartz Filter Experiments

Executive Summary

Quartz filter experiments were recently conducted at the USEPA National Air and Radiation Environmental Laboratory (NAREL) located in Montgomery, Alabama, as part of the continuing quality assurance oversight for the $PM_{2.5}$ Speciation Trends Network (STN). These experiments were designed to further our understanding of the undesirable contamination that may be transferred from a [filter holder] cassette to the filter. These experiments utilized the Thermal/Optical Transmittance (TOT) technique approved for the STN to measure Organic Carbon (OC) and Elemental Carbon (EC) captured from the ambient air. During this study, several cassette materials were tested for their potential to contaminate a quartz filter. The following cassette materials were examined: Delrin®, ABS/Polycarbonate (blue/poly), Ertalyte®, and Teflon®.

This study shows that measurable OC contamination can be transferred from some cassette materials to a blank quartz filter. Previous studies using gravimetric measurement have shown that some cassettes will contaminate a Teflon® blank. The gravimetric measurements, however, were not able to determine specifically where on the Teflon® filter the contamination was located, nor were they able to determine the chemical species present in the contamination. This study shows that the level of OC contamination present at the perimeter of a circular quartz filter is higher than the level found within the central region of the filter. This discovery is not surprising since the perimeter of the filter comes into direct contact with the plastic cassette. Normally the perimeter of a quartz filter is not analyzed because it is outside the deposit zone for captured PM. But for this study, special effort was made to determine specifically where on the filter the unwanted contaminants were located. As a result of this study, it is now possible to understand how Teflon® blanks may show contamination from faulty cassettes much sooner than quartz blanks from the same events. Only a portion of the contamination transferred from the cassette to a quartz filter is actually measured during the TOT analysis whereas one hundred percent of the contamination transferred to a Teflon® filter is measured during the gravimetric analysis.

History and Background

A series of experiments were performed in the summer of 2001 to investigate a large number of contaminated field and trip blanks collected by the STN. Most of the blank failures were from gravimetric mass measurements and not from the TOT carbon measurements. These early experiments demonstrated that some [filter holder] cassettes, especially new ones supplied by Met One and made of Delrin® plastic, will transfer a significant amount of contamination to a blank Teflon® filter as revealed by gravimetric mass measurements.[1] Early experiments also showed that the contamination received by a Teflon® filter was permanent, and the contamination was not removed from the filter as the ambient air passed through the filter during sample collection.[2] One or more organic compounds released from the cassette plastic were suspected to cause the blank contamination, but experiments had failed to identify the chemical(s) responsible.[3] The investigation has now been expanded to examine quartz filters. A quartz filter is used by the STN to capture PM for subsequent carbon analysis. Recent experiments described in this report were conducted to measure the level of contamination which might be transferred from the cassette to a quartz filter.

Previous studies have shown that the level of contamination observed on the Teflon® filter depends upon three main factors: (1) the length of time given to direct contact between the filter and the cassette, (2) the temperature of the cassette during the period of contact, and (3) the intrinsic contamination potential of the individual cassette. It was assumed that these principles are also true for quartz filters. For this study, a quartz filter blank was mounted into the test cassette, and the cassette was assembled into a sampler module which is the current practice for STN field and trip blanks. All modules were placed outside in the Montgomery autumn weather for two weeks before they were brought back inside and disassembled for analysis of the filter. Only new cassettes were tested since previous studies have shown that the contamination potential of an individual cassette decreases with use.

The TOT analysis normally proceeds by punching a sub-sample from the central region of the quartz filter. The sub-sample must be small enough to fit into the narrow oven of the TOT instrument. The instrument provides results expressed as micrograms of carbon per square centimeter of filter (:g/cm^2). Instrument results may be converted to ambient air concentration using Equation 1.

$$\text{ambient air concentration} = \frac{\mu g}{cm^2} \times \frac{\text{filter deposit area}}{\text{volume of air sampled}} \qquad \text{Eq. 1}$$

Multiple punches were taken from each test filter to locate where on the filter the contamination was highest. TOT measurements for carbon were taken not only from the central portion of the quartz filter, as required for STN samples, but also from the perimeter of the filter which makes direct physical contact with the cassette. Examination of the filter perimeter was critical for this study because the routine analysis does not account for any carbon present on the outermost 4-mm region of the circular filter. This 4-mm perimeter makes direct contact with the plastic cassette and is therefore unavailable for ambient air PM deposits. A custom punch device shown in Figure 1 was created to optimize taking punches from the perimeter of the

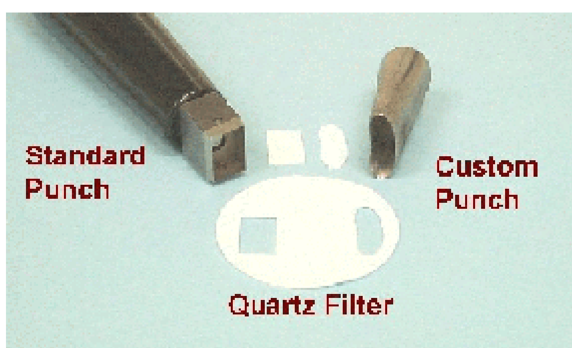

Figure 1

circular filter. The standard rectangular punch device, also shown in Figure 1, was not useful for accurate analysis of the filter perimeter.

Experimental

Each experiment was designed to mimic a travel blank exposed to outside weather conditions in Montgomery, Alabama, during the autumn. The instrumentation and the analytical method used at NAREL for this study were essentially the same as those used at the Research Triangle Institute (RTI). RTI is the prime contractor for STN samples.

Several cassette materials were tested during this study. Delrin® cassettes were obtained from two suppliers: Met One and Anderson. Delrin® is a polyacetal Dupont plastic. Ertalyte® cassettes were also supplied by Anderson. Ertalyte® is a Polyethylene Terephthalate Polyester plastic registered to DSM Engineering. The blue/poly cassettes which are constructed from an ABS/Polycarbonate plastic were supplied by R&P. The Teflon® [filter holder] cassettes were supplied by URG, and Teflon® is a Polytetrafluoroethylene Dupont plastic. Duplicate experiments were performed to test each cassette material.

Each new cassette was cleaned with PREMOISTENED CLEAN-WIPES™ (VWR Scientific 21910-111) using RTI's standard procedure, and allowed to air dry. New sampler modules were also cleaned using the wipes. A blank quartz filter was mounted into each new cassette, and the cassette was assembled into a sampler module. Each final assembly resembled a routine sealed STN trip blank except for one modification: each assembly contained an extra blank quartz filter which was not mounted into a cassette, but was folded, wrapped loosely in aluminum foil, and placed inside the module near the inlet. This extra "module filter blank" was designed to monitor contamination that did not require direct contact with the test cassette.

The modules were placed into a thermally insulated cooler along with a Dixon data logger which continuously recorded the humidity and temperature inside the cooler. No ice or other heat sink materials were placed into the cooler. The cooler was placed outside the building under a canopy to offer shade and protect it from rain. After two weeks the cooler was brought back into the laboratory, and the modules were disassembled to recover the filters for analysis.

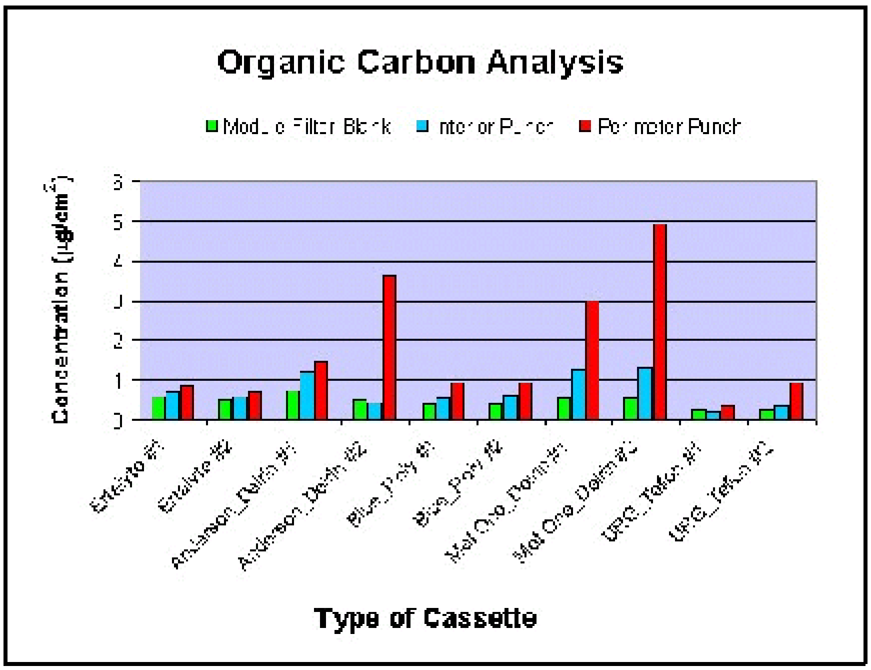

Figure 2

Results for the outside test period of November 16-30 are summarized with a bar graph in Figure 2. For each cassette tested, the graph shows the OC concentration found on the interior punch, the perimeter punch, and the module filter blank. For three of the Delrin® cassettes tested, the OC contamination was more than 2 :g/cm^2 on the perimeter of the test filter. For every cassette tested, the OC contamination on the perimeter punch was at least slightly higher than the contamination on the interior punch. Table 1 at the end of this report contains the numerical data which are graphically presented in Figure 2. Elemental carbon is not reported because it was not detected during the study.

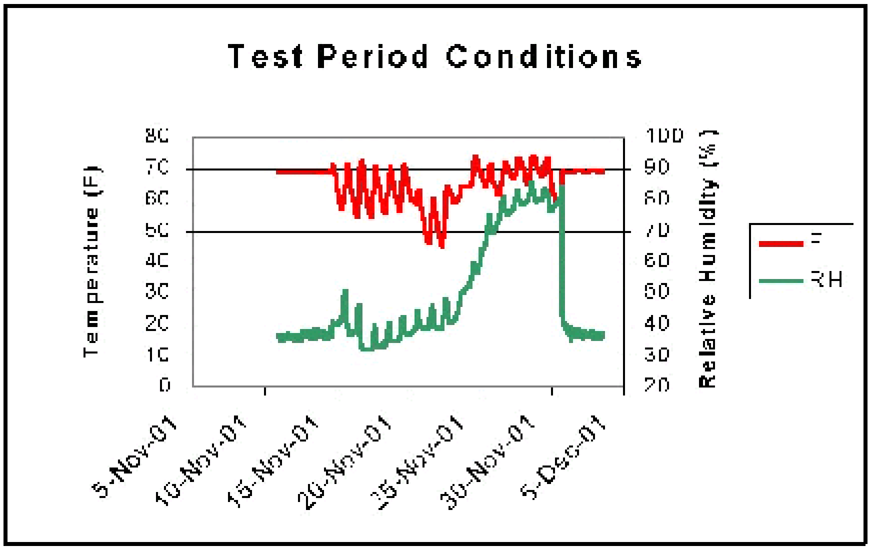

Figure 3

Outside temperature and humidity conditions for the test period are presented in Figure 3. Humidity was high during the last week of the test period because of rain. Daytime temperatures were usually in the lower 70's with temperature decreasing during the night.

Conclusions

This study has examined several cassette materials for their potential to contaminate a quartz filter. Five cassette materials were tested in duplicate under conditions that are typical for STN trip blanks. Delrin® was the material which showed the greatest level of contamination, and the Delrin® cassettes were supplied by two different vendors.

This study has shown that a faulty cassette will contaminate a quartz filter, and the contamination is more pronounced at the perimeter of the filter. Teflon® filter blanks are out-of-control if contamination on the filter exceeds a mass of 30 :g. This control limit applied to a quartz filter will represent approximately 2.5 :g/cm^2 as determined by the TOT analysis.

30 :g per 11.76 cm^2 of <u>interior</u> filter deposit area = 2.5 :g/cm^2

None of the interior punches analyzed during this study exceeded the 2.5-:g/cm^2 control limit. Therefore a routine analysis of all the test filters would not have signaled a problem. The out-of-control levels observed for three of the perimeter punches would never have been measured for a routine STN sample. Perimeter punches were analyzed during this study for only one purpose: to determine where on the test filter undesirable contaminants were deposited.

References

1. Technical Memorandum: "Evaluation of MetOne SASS Field and Trip Blank Issues". U.S. Environmental Protection Agency. National Air and Radiation Environmental Laboratory, Montgomery, AL. July 2001.

2. Technical Memorandum: "Experiments with Delrin® Cassettes in a MetOne SASS Sampler". U.S. Environmental Protection Agency. National Air and Radiation Environmental Laboratory, Montgomery, AL. August 2001.

3. Gutknecht, W.F., J.A. O'Rourke, J.B. Flanagan, W.C. Eaton, M.R. Peterson, and L.C. Green. "Research to Investigate the Source(s) of High Field Blanks for Teflon® PM2.5 Filters", EPA Contract 68-D99-013. Research Triangle Institute, Research Triangle Park, NC. July 2001.

TABLE 1

Cassette Description	Module	OC Concentration (:g/cm^2)		
		Module Filter Blank*	Test Filter Interior Punch	Test Filter Perimeter Punch
Ertalyte® #1	RASS #1	0.55	0.68	0.83
Ertalyte® #2	SASS #9	0.50	0.58	0.68
Anderson Delrin® #1	RASS #2	0.71	1.18	1.46
Anderson Delrin® #2	SASS #9	0.50	0.41	3.62
Blue/Poly #1	SASS #6	0.38	0.51	0.93
Blue/Poly #2	SASS #6	0.38	0.63	0.93
Met One Delrin® #1	SASS #7	0.52	1.25	3.01
Met One Delrin® #2	SASS #7	0.52	1.32	4.93
URG Teflon® #1	MASS #1	0.24	0.18	0.36
URG Teflon® #2	MASS #1	0.24	0.34	0.89

*The blank filter was placed into the module but was not mounted into a cassette.