ETV Joint Verification Statement

TECHNOLOGY TYPE:  Personal Cascade Impactor Sampler
APPLICATION:  Personal Air Sampling
TECHNOLOGY NAME:  Sioutas Personal Cascade Impactor Sampler with Leland Legacy® Pump
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The U.S. Environmental Protection Agency (EPA) has established the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies. Information and ETV documents are available at www.epa.gov/etv.

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The Advanced Monitoring Systems (AMS) Center, one of six technology areas under ETV, is operated by Battelle in cooperation with EPA’s National Exposure Research Laboratory. The AMS Center evaluated the performance of the Sioutas Personal Cascade Impactor Sampler (PCIS) with the Leland Legacy® Pump. This verification statement provides a summary of the test results.
VERIFICATION TEST DESCRIPTION

The objective of this verification test is to provide quantitative and qualitative performance data on the Sioutas PCIS and the Leland Legacy® pump under a range of typical and extreme operating conditions. To meet this objective, the verification test for the Sioutas PCIS and Leland Legacy® pump was conducted by Battelle from January 16, 2006 through February 7, 2007. The longer than normal period of testing resulted from the necessity to repeat the pump testing on pumps that were newly retrofitted by the manufacturer to improve performance under extreme operating conditions.

The performance of the PCIS was evaluated during a short-term (10-15 minute) sampling of a test aerosol (potassium chloride) generated in a large chamber (17.3 m³), and a long-term (48 hour) sampling of ambient air particulate. Three test aerosols were generated for 10 or 15 minutes, resulting in average total particulate matter (TPM) concentrations of circa 3 mg/m³. During these tests, chemical and gravimetric comparisons were made of the PCIS’s sampling efficiency relative to three more well-known “reference” personal and fixed-site air samplers that were collocated with the PCIS. The short-term sampling efficiency testing compared the gravimetric results for samples collected using the test Sioutas PCIS to those from the reference samplers: Delron Cascade Impactor (DCI-6, 0.5, 1.0, 2.0, 4.0, 8.0, 16 µm cutpoints), Federal Reference Method PM₂.₅ (FRM, 2.5 µm cutpoint), and Personal Environmental Monitor (PEM, 10 µm cutpoint). The long-term sampling efficiency testing compared the ambient air particulate metals levels, measured using x-ray fluorescence (XRF) spectroscopy, in samples collected by collocated PCIS and reference PEM (2.5 µm cutpoint) samplers. Although the focus of the test was on characterizing metals in ambient air, we have included data in the discussion that follows from other non-metallic elements that were also observed. Note: of the reference samplers chosen for comparison, the FRM PM₂.₅ is the only true reference sampling method. In order to evaluate the Sioutas PCIS’s smaller cutpoints individually and as a whole, we also included two other samplers, the DCI-6 and the PEM, that are well characterized in the literature and have similar aerodynamic cutpoints and flow rates.

A human subjects study was also conducted to evaluate more subjective sampler parameters, including the PCIS’s ease of use and subject acceptance. Seven non-smoking subjects wore the PCIS for a period of 48 hours and kept a simple time/activity diary. At the end of the sampling period, subjects completed a questionnaire about the PCIS’s ease of operation, reliability, and their acceptance of the device. The pumps were equipped with hidden multidirectional accelerometers that logged the pump’s frequency and intensity of movement. Examination of the accelerometer data and the subject’s time/activity diary provided a semi-quantitative measure of each subject’s compliance with the air sampling protocol.

The Leland Legacy® pump was evaluated for its duration of operation on a single battery charge while sampling at different pressure drops, and its 24-hour performance in extreme temperature and humidity environments. Inter- and intra-pump variations in flow rate, sound level, and duration of operation were measured. During the initial pump testing, half of the pumps failed. A failure analysis was performed by the vendor, the problem was identified, and the original pumps were retrofitted with a specially designed hexagonal pin to prevent future failure. All pump testing was repeated on the retrofitted pumps in order to compare the performance of the original and retrofitted pumps. All test results from both the original and retrofitted pumps are included in this report.

QA oversight of verification testing was provided by Battelle and EPA. Battelle QA staff conducted a technical systems audit, a performance evaluation audit, and a data quality audit of 10% of the test data.

This verification statement, the full report on which it is based, and the test/QA plan for this verification test are all available at www.epa.gov/etv/centers/center1.html.
TECHNOLOGY DESCRIPTION

The following description of the Sioutas PCIS and the Leland Legacy® pump is based on information provided by the vendor. This technology description was not verified in this test.

The Sioutas PCIS is designed to separate and collect airborne particulate matter (PM) in specific size ranges, generally referred to as coarse, fine, and ultrafine. Results of the micro-environmental sampling performed with the Sioutas PCIS can be used to characterize particle mass, particle size, and chemical composition (constituents) of PM pollutants in air. The Sioutas PCIS was designed to be operated with the Leland Legacy® pump, which allows for 24-hour sampling on battery power.

The Sioutas PCIS consists of four impaction stages (each with a collector plate and an accelerator plate) followed by an after-filter. Each impaction stage holds a 25 mm (0.5 µm pore size) impaction substrate that collects particles above a specific size (50% cut-point) starting from the largest particles with aerodynamic diameters >2.5 µm, Stage A; and going smaller: 2.5 – 1.0 µm, Stage B; 1.0 – 0.5 µm, Stage C; and 0.5 – 0.25 µm, Stage D. The smallest particles (<0.25 µm) collect on a 37-mm (2.0 µm pore size) after-filter. The Sioutas PCIS, which is clipped onto a shirt lapel or pocket, is connected with tubing to a calibrated Leland Legacy® constant flow sample pump that draws air through the impactor at 9 L/min. The 9 L/min flow rate must remain constant for precise particle separation. The chemically inert Teflon® impaction substrates and after-filter recommended for use in the Sioutas PCIS are suited for both gravimetric analysis of PM mass and chemical analyses of PM constituents.

Extended operation of the pumps at higher than specified backpressures was the cause of the failures experienced during testing. The increased backpressure was great enough to break the crank-arm pivot pin loose from the diaphragm yoke assembly. SKC designed a new hexagonal-shaped pin head to ensure that this condition would not occur. The new pin design will be incorporated in Leland Legacy® pumps manufactured by SKC.

The Sioutas PCIS is 3.4 inch tall by 2.2 inch in diameter (9.0 by 5.6 cm); its inlet is 3/8-inch outer diameter (0.95 cm), ¼-inch inner diameter (0.63 cm); and its outlet is 3/8-inch outer diameter, ¼-inch inner diameter. It weighs 5.6 ounces (159 grams). The price of the Sioutas PCIS is $505 and the Leland Legacy® pump costs $1,249. Together the pump and PCIS costs $1,754. Additionally, impaction substrates are approximately $195 per 100 (each sampling requires four of these), and 50 collection filters cost approximately $230 (each sampling requires one of these).

VERIFICATION RESULTS

Pump Testing: Several of the original pumps failed to sample for at least 24 hours when sampling under 15 inch H₂O (3 of 12) and 19 inch H₂O (5 of 12) pressure drops. All of the original pumps sampled under an 11 inch H₂O pressure drop via battery power for 28-34 hours in a moderate temperature and humidity (25°C and 30%) environment after a 15-hour battery charge. Due to repeated pump failures during the pressure drop testing, the original pumps were not evaluated at high temperature-moderate humidity (40°C-60%) and high temperature-high humidity (40°C-90%) sampling environments. Under a 19 inch H₂O pressure drop overall sampling duration was 9-10 hours fewer than under an 11 inch H₂O pressure drop, and average sound levels increased by 2.1 dB. The average duration of operation for the original pumps deviated by 5.6 – 29% during these tests. Prior to shutdown from battery drain, the average flow rates during these tests never deviated by more than 5.0% for the original pumps.
Under all conditions, the retrofitted pumps maintained flow rates that were within 10% of the manufacturer’s recommended values and sampled for at least 26 hours. All of the retrofitted pumps sampled for 31-35 hours in a moderate temperature and humidity (25°C and 30%) environment, and for at least 24 hours in both high temperature-moderate humidity (40°C-60%) and high temperature-high humidity (40°C-90%) environments. When sampling under a 19 inch H2O pressure drop, the retrofitted pumps operated for 26-28 hours, which was 6-7 fewer hours than under an 11 inch H2O pressure drop, and sound levels increased 4.3 dB. The sound level of a single pump operating in the noise-reducing jacket ranged from 48-64 dB, a sound level that is on par with normal conversation. The average duration of operation for the retrofitted pumps deviated by less than 2.6% for the 11-19 inch H2O test. Prior to shutdown from battery drain, the average flow rates during these tests never deviated by more than 2.1% for the retrofitted pumps.

**Sampling Efficiency Comparison:** Sampling efficiency of the impactors was gravimetrically evaluated for PM$_{2.5}$, TPM, and individual impaction stages, as appropriate, by sampling a test aerosol generated in a large environmental chamber. The aerosol concentrations generated for the sampling efficiency test were, although optimal for obtaining gravimetrically measurable levels of particles on all stages of the Sioutas PCIS, several orders of magnitude higher than those experienced in most real-world settings. These aerosol concentrations caused the PEM pumps to operate at backpressures that were greater than the manufacturer’s specifications, although the pump logs did not report any pump failure. Because the upper particle loading limit is a complex function of the ambient particle size distribution and type, humidity, individual filter used, capacity of the sampler flow rate control system, and possibly other parameters, it is not known whether these high concentrations resulted in particle bounce and/or affected the cutpoints of the particles collected for the DCI-6 and PEM samplers. The particle loadings did not exceed the Sioutas PCIS manufacturer’s operating specification for maximum particle load, 3.16 mg/stage, nor did they exceed the federally mandated capability of the FRM, 4.8 mg PM$_{2.5}$/filter. The humidity in the environmental chamber did not exceed 29% and the temperature did not increase by more than 1.7°C during the one hour sampling. The samplers performed consistently, as the inter-sampler variability for all samplers in each of the three tests did not exceed 11% for the FRMs, 4% for the PEMs, 15% for the DCI-6, and 10% for the PCISs for each cutpoint in which the gravimetric masses were above the method detection limit.

For TPM collected, Sioutas PCIS data show a negative bias of 15% compared to the DCI-6 and 17% compared to the PEM data. When comparing particles in the ≤0.5 µm size range, the PCIS data show a negative bias of 35% compared to the DCI-6 data. For slightly larger particles in the 0.5 – 1.0 µm size range, the trend is reversed: the Sioutas PCIS data show a positive bias of 26% compared to the DCI-6 data. The PCIS PM$_{2.5}$ results show a negative bias of 16% compared to the data for both of the reference samplers, including the DCI-6, which was collecting particles that are only ≤2.0 µm in diameter.

**Sampling Metals in Ambient Air:** When the XRF analytical results of the PM$_{2.5}$ collected using the Sioutas PCIS samplers are compared to those collected using the PEM samplers, the dataset of S, Cl, Si, Ca, Fe, K and Zn results falls along a line that is significantly statistically different from the unity line, although the y-intercept, -0.54, is not significantly different from zero. The majority of the data falls below the unity line indicating an overall negative bias, 36%, for the Sioutas PCIS results as compared to those for the reference PEMs. This negative bias is similar to that found in the test aerosol high concentration result discussed in the sampling efficiency comparisons, above. At both concentration levels, sub-µg/m$^3$ of metal/element versus mg/m$^3$ of particulate, the Sioutas PCIS results show a negative bias compared to the data for the reference samplers.

Results near the method detection limit (MDL) showed the highest negative bias (74%). Because the PM$_{2.5}$ particles are collected onto four separate stages for the Sioutas PCIS, if the level collected on each stage is at or near the MDL, results may not correlate well with those from a single stage PEM impactor that collects all of the PM$_{2.5}$ onto one filter.

Also, the slit-like accelerator plate nozzles in the Sioutas PCIS generated thin deposition patterns on the sampling substrates which made accurate quantitative analysis of the metals/elements by XRF difficult.
Because of the shape of the deposition pattern, analysis by ICP-MS is recommended over XRF, although this technique would require an additional wet chemical extraction step.

**Ease of Use, Reliability and Subject Acceptance/Compliance:** Questionnaire responses showed that subjects felt strongly that the PCIS noise was too loud and that they did not like wearing the pump; however, the noise wasn’t loud enough to prevent them from thinking or talking on the phone while wearing the sampler. They also felt strongly that the sampler (Leland Legacy® pump and Sioutas PCIS combination) was easy to take on and off and plug into the wall to run on A/C power while they slept. Examination of the accelerometer and time/activity diary data showed that although all of the subjects said they followed the sampling protocol, only half of the subjects followed the 48-hour sampling protocol; the other half stopped wearing the pump roughly after the first 24 hours.

A Battelle laboratory technician’s comments indicate that the Sioutas PCIS’s plastic filter retainers require quite a bit of force to place them flush on the impaction plate. It is important that this be accomplished, because if the retainers are not completely flush to the plate, the pressure drop of the impactor exceeds 16 inch H₂O. The majority of the subject recommendations included reducing the Sioutas PCIS’s noise level.

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