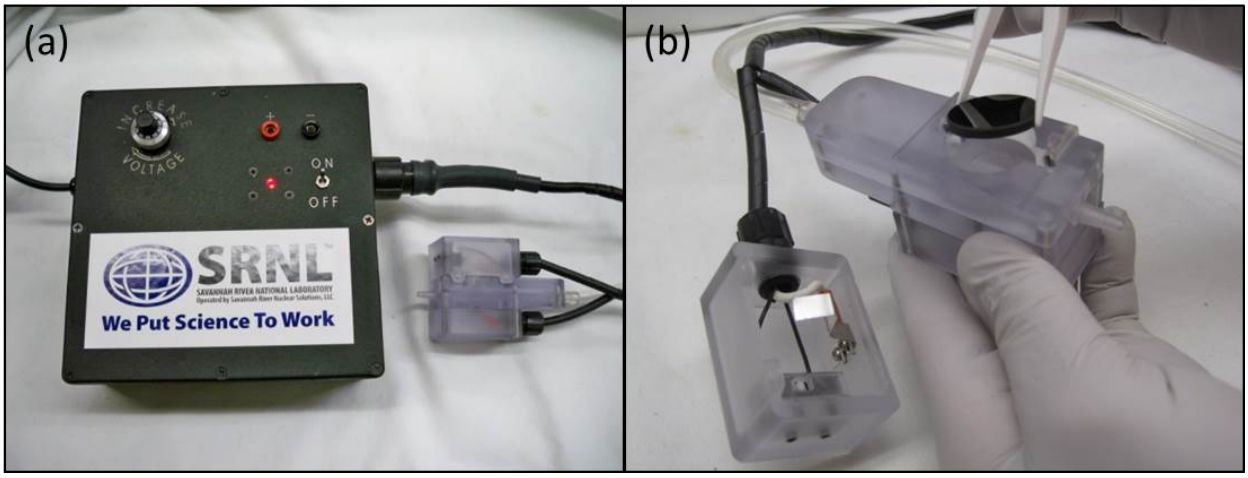
Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

Validation Study of the SRNL Vacuum Aerosol Contaminant Extractor

SRNL recently developed a prototype device for the International Atomic Energy Agency (IAEA) to prepare particulate samples collected on swipes for laboratory analysis. The Vacuum Aerosol Contaminant Extractor (VacACE) utilizes electrostatic precipitation in lieu of the impaction or ultrasonic solvent extraction methods presently employed by the IAEA to place particles of interest on carbon planchets for investigation. The project was funded by the Intentional Safeguards Projects Office (ISPO) with scope for device design and fabrication, but no scope for validation or testing. Without documented validation of the tool, sample processing and subsequent analysis fidelity cannot be assured. A defensible validation study needed to be performed along with the development of a standard protocol for the VacACE to become an accepted sample process method for DOE and WFO needs. The present effort determined collection efficiancy, demonstrated a proof of concept with standardized particulates, and produced a validated VacACE sampling protocol.



(a) VacACE controller and electrostatic unit together; **(b)** VacACE electrostatic unit open with vitreous carbon planchet being removed

Awards and Recognition

None

Intellectual Property Review

This report has been reviewed by SRNL Legal Counsel for intellectual property considerations and is approved to be publically published in its current form.

SRNL Legal Signature

Signature **Date**

LDRD-2015-00076
LDRD Report

Validation Study of the SRNL Vacuum Aerosol Contaminant Extractor

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Thrust Area: ST1

Project Type: Quick Hit

Project Start Date: May 1, 2013
Project End Date: September 30, 2014

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FY2015 Objectives

- Determine the VacACE efficiency for collecting particles from swipes
- Optimize VacACE parameters (i.e., corona voltage, flow rate, dwell time, etc.)
- Develop an operational protocol for sample acquisition

Introduction

Subsampling from swipes taken in nuclear facilities is a standard NWAL/IAEA practice which typically utilizes vacuum impaction. The VacACE provides a needed alternative to vacuum impaction methods because they yield uneven particle loading and require the use of an adhesive known to complicate subsequent analytical techniques. In particular analysis of samples prepared by the impaction method can be use a sticking agent (e.g., polyisobutylenes [PIB] in nonane) to improve particle retention. Unfortunately the sticking agent changes the distribution of particles collected on the planchet, induces charging effects that interfere with SEM and SIMS measurements, and elevated temperatures required for removal of the adhesive may cause undesirable changes in the planchet surface and/or particle chemistry. The VacACE eliminates such complications because it does not require an adhesive to improve sample retention and sample analysis can be performed directly on collection surfaces with minimal additional steps for sample preparation. Successful validation testing would allow SRNL to utilize this improved sample processing capability and subsequently benefit from the inherent overall enhancement in analytical results.

Approach

The VacACE allows a user to adjust both the power to the electrostatic collector (i.e., the corona voltage) and airflow into the device. The first goal was to determine collection efficiencies for particles across a broad range of particle sizes as a function of applied voltage and flow rate. A simple method to determine collection efficiency is to compare the number of particles exiting the device with and without the device activated. Additional efforts included measuring the particle extraction efficiency by comparing regions of a swipe with and without VacACE application with standard microanalytical

methods. A standard operational protocol was distilled from these measurements which optimizes collection efficiency and sample processing time.

Results/Discussion

Collection efficiencies were determined by counting the number of airborne particles exiting the device with and without an applied corona voltage. Collection efficiencies were found to be as high as 65% for 5 micron particles, and 52% for 300 nm particles; slightly better than the impaction method presently employed by the IAEA (approx. 50%). Additionally, collection efficiency can be tailored by adjusting the corona voltage. Using automated SEM analysis, the entire collection region was scanned to determine the particle distribution on the collection surface.

FY2015 Accomplishments

- Determined VacACE efficiencies for collecting particles from swipes:
  - 52% for 300 nm particles
  - 60% for 500 nm particles
  - 65% for 5 micron particles
- Automated SEM scanning/analysis was used to determine sample distribution on the collection surface
- Developed a sampling protocol for collecting particles from sample swipes

Future Directions

Completion of this study validated the VacACE resulting in enhanced analytical capabilities directly applicable to future needs. Validated VacACE capabilities will be included in and enhance future proposals to DOE and WFO customers.

FY 2015 Publications/Presentations

None

References

None

Acronyms

IAEA – International Atomic Energy Agency
ISPO – International Safeguards Projects Office
NWAL – Network of Analytical Laboratories
SRNL – Savannah River National Laboratory
VacACE – Vacuum Aerosol Contaminant Extractor

Intellectual Property

None

Total Number of Post-Doctoral Researchers

None