

## Use of an Automated SEM to Detect Laboratory Contamination

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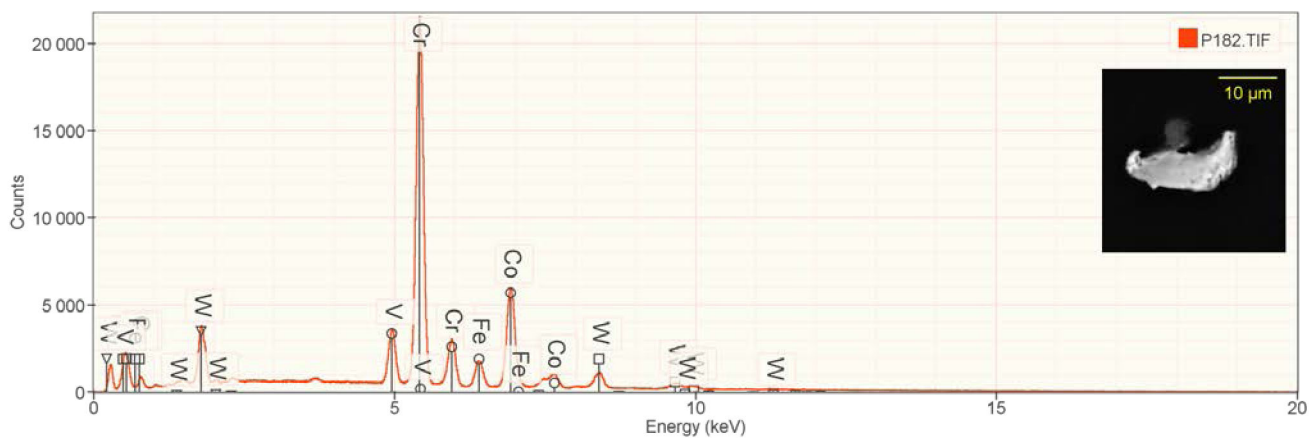
Automated SEM analysis can be used to characterize a large number of particles in a relatively short time. Since there is minimal sample preparation, it provides a quick screening method to identify sub-populations of compositionally distinct particles. For problems in which the abundant classes provide the critical answer, possible contamination at the trace level is of little interest. However, for those problems in which the trace constituents are significant, it is critical to characterize the laboratory background and to measure blanks as part of the analytical strategy to avoid ambiguity that can be introduced from contamination sources.

Contamination may be introduced at any point in the sample handling process, but passage through general purpose laboratories can be sources of unexpected materials. To explore our local contamination sources in such a situation, we collected a total of 30 mounts at various locations around a laboratory that is currently used as a machine shop. It has a variety of instruments such as grinders and polishers. Sampling was done from a variety of locations including the top of a service strip along the wall, horizontal and vertical surfaces of instruments as well as bench tops, the floor, and walls. We intentionally collected from both horizontal and vertical surfaces in hopes of collecting a wide variety of particle sizes. Shards and large particles that extended off the edges of the sticky tab were removed from the mounts. Particles were collected on a Gun Shot Residue (GSR) mount, which is a carbon-coated SEM stub in a plastic case. We added a carbon sticky tab to provide a smooth surface and cut three fiducial marks around the edges of the mount to aid in particle relocation. The samples were analyzed without coating. The analysis was done on an ASPEX Personal SEM<sup>1</sup> using the Automated Particle Analysis routine using the backscatter detector at 250 × and 25 keV with a 3 second EDS analysis. The data was analyzed using standards with the proprietary NIST Graf particle analysis program and DTSA II [1]. Both programs are based on the same spectrum quantification algorithm library. The Graf program does an unbiased assessment of the particle classes present, which can then be refined by defining “rules” that examine associations of elements.

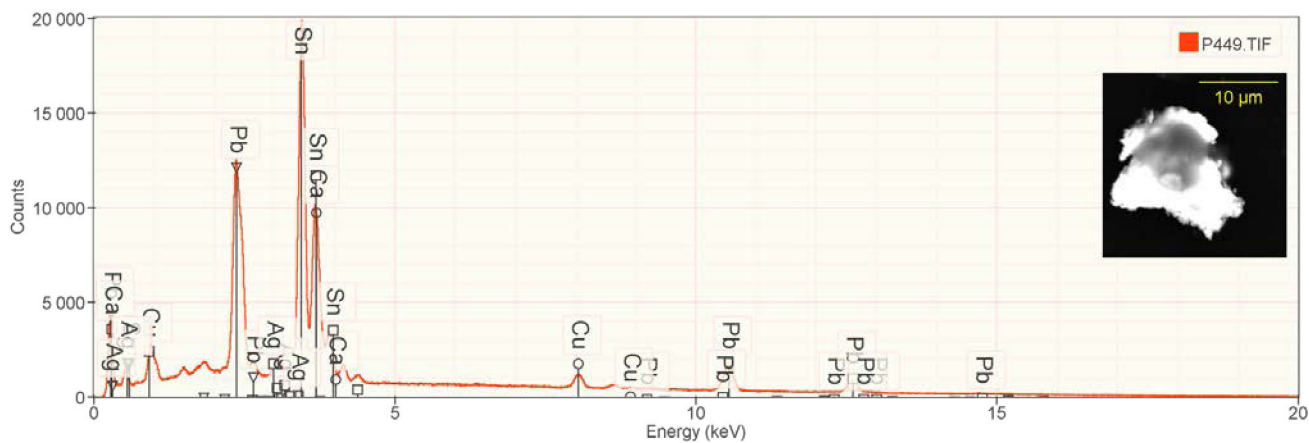
The analysis revealed dominant classes that contained thousands of particles including Al-rich, Fe-rich, stainless steel (Fe, Ni, and Cr), Ti-containing particles, silicates, aluminosilicates, carbon-rich, and other particles typical of soil or dust or materials-processing expected in a machine shop. In addition, there were several particle classes where we found a single particle or a few particles. Some of the more unusual particles contained Co in various elemental mixtures (Figure 1), a C e-containing particle, numerous particles containing large amounts of Sn (Figure 2), and a shard of ZnO. By establishing a database of the dominant as well as the rare particles that form the laboratory background and measuring blanks that accompany each working sample, contamination can be recognized.

[1] Ritchie, N., “Getting Started with NIST DTSA-II”, *Microscopy Today*, (19) 2011 26-31; DTSA-II is available for free download at [www.cstl.nist.gov/div837/837.02/epq/dtsa2/index.html](http://www.cstl.nist.gov/div837/837.02/epq/dtsa2/index.html)

<sup>1</sup> Certain products, either public domain or commercial, are identified in this talk. Identification of such products does not imply recommendation or endorsement by NIST, nor does it imply that the identified product is the best available.



**Figure 1.** Image and Spectra of a Cr, Co, W, V particle



**Figure 2.** Image and Spectra of a Sn, Pb particle