The rapid screening of particulate samples is critical in confirming or denying the presence of elements of interest. Typically, high atomic number particles found on waste air filters or precious metals within minerals require a complex dissolution and chemical analysis to detect and quantify them, without giving any information on size or shape morphology. To measure their size or shape morphology, electron microscopy is required separately from the dissolution. However, secondary electron microscopy-based energy dispersive spectroscopy is limited to the top 1 μm to 5 μm of the sample; using this technique to interrogate particles below the sample surface typically involves destructive methods, such as focused ion beam (FIB) ablation. Micro X-ray fluorescence spectroscopy (micro-XRF) instruments, while able to detect sub-surface particles, are limited in that they cannot easily distinguish surface from sub-surface particles. The depth discrimination limitations of micro-XRF can be overcome by confocal micro-XRF which allows for the spatial discrimination of X-ray fluorescence signals in all three spatial axes and allows for the detection and identification of the elements present at depth. Fluorescence data is acquired by rastering the sample, acquiring one voxel at a time; hence, large samples can require days or even weeks to fully acquire a 3D elemental image. This analysis time can be significantly reduced by pre-screening the samples for X-ray absorption values of interest by utilizing a complementary, non-destructive X-ray imaging technique: micro X-ray computed tomography (micro-CT). This technique rapidly collects absorption contrast images of a sample and reasonable 3D data sets can be acquired within a few hours.

In this presentation, we will give an overview of the qualitative elemental analysis of particles composed of high atomic number elements (high-Z) embedded within a mineralogical sample using two laboratory-based X-ray techniques: micro-CT and confocal micro-XRF. The sample, composed of pressed mineral particles within a low-Z matrix, was first imaged non-destructively using micro-CT. Two particles of interest were identified from the tomogram based on regions of high X-ray attenuation, indicating the presence of high-Z particles of interest. One surface particle was identified, in addition to one subsurface particle which was located ~300 μm below the sample surface. These particles were then analyzed and elementally identified using confocal micro-XRF spectroscopy. By coupling these two techniques, we were able to screen the mineralogical sample for high-Z particles of interest and identify their elemental composition in a non-destructive manner.