

## **XRD and XRF**

1. Both XRD and XRF are spectroscopic techniques dependent on the interactions between x-rays and the sample being analyzed. XRD is a destructive method which records the diffraction patterns of the x-rays, and XRF is a non-destructive technique which records the energies or wavelengths of secondary x-rays emitted by the excited sample. Spectra generated from both methods are compared to sets of standards to identify what elements or crystals are present. While XRF spectra reveal the chemical make-up of a sample, XRD provides information about the crystalline microstructure and is able to distinguish between different forms of compounds with the same elements. Elements ranging from either beryllium or sodium up to uranium can be detected by XRF in concentrations down to parts per million. XRD can be used quantitatively to determine relative percentages of different crystal phases present.
2. XRD is only useful in the identification of crystalline materials, including lithics, metals, and ceramics. Amorphous compounds and some organic materials do not form the highly ordered lattices of crystals, and cannot be detected with XRD. These include plastics, fibers, dyes, and glasses. However, it is important to note that XRF can be used in all of these instances, and XRD could be implemented in the analysis of certain opaque glasses and glazes containing crystal phases of compounds other than amorphous silica.
3. The makers of handheld XRF devices advertise that they can perform elemental analysis regardless of a sample's size, shape, or location.<sup>1</sup> However, samples for XRF and XRD must be at least comfortably larger than the x-ray beam itself—roughly larger than 1 cm<sup>2</sup>. Samples prepared for bench-top analysis must be able to fit inside the machinery, so should be at most about 15 cm<sup>2</sup>.

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<sup>1</sup> Henseler, J. "Take the Laboratory with You: Portable XRF." *American Laboratory* **2009**, 41(2), 48-49.

## SEM-EDS

1. SEM and EDS, straightforwardly referenced in their names, are electron microscopy and x-ray spectroscopy techniques, respectively. SEM allows for non-destructive viewing of highly magnified images of a material, clearly displaying surface textures and characteristics. EDS provides additional information on elemental make-up.
2. As SEM magnifies up to 50,000x, samples on the order of microns may be examined. Larger samples must be able to fit inside the vacuum chamber, which can be as large as 5x3cm. Any shape of object can be used as long as it fits in the chamber, however flatter surfaces are preferable and easier to analyze. There are no limitations in terms of the type of material which may be used, however additional preparative measures may be necessary (see #4, 6).
3. The detection limit of EDS, for elements ranging from beryllium to uranium, can be as low as 0.1 atom percent, and can vary up to a few atom percentages depending on the element.
4. Samples which are not conductive must be coated with a thin layer of gold, graphite, or platinum. Some metals, in order to be able to view grain textures via SEM, must be etched.
5. SEM-EDS is generally a non-invasive, non-destructive technique. This is true unless the specimen in question is too large to fit in the SEM chamber and a smaller sample must be removed, or if the samples must be coated or etched.
6. Basically all types of materials may be examined with SEM-EDS, although some may require extra preparation steps as discussed.
7. Secondary electrons (SE) provide surface texture images and back-scattered electrons (BSE) show compositional heterogeneity by showing the average atomic number in an area. EDS, akin to XRF, reveals relative elemental composition.