

# Bulk sample characterisation and surface analysis

Techniques such as simultaneous Proton-Induced X-ray Emission (PIXE), Proton-Induced Gamma-ray Emission (PIGE) and Rutherford Back-Scattering (RBS) allow bulk element determinations to parts per million concentrations, and depth profiling of surface layers.

Samples are measured in vacuum and measurement of samples can be automated. The range of analyses possible covers elements from hydrogen to uranium

Applications include pigment composition of paintings; studies of Aboriginal Australian and Polynesian artefacts to identify the origin of samples and migratory history; and composition of geological samples as an aid to mineral ore prospecting. This service includes spectrum evaluation and data interpretation.

Please discuss your proposal with the appropriate ANSTO Contact Scientist before submitting your proposal as they will assist you in making the correct capability selection.

## Capability Selections

- Particle Induced X-ray Emission spectrometry (PIXE)
- Proton-Induced Gamma-ray Emission spectrometry (PIGE)
- Proton Elastic-Scattering Analysis (PESA)
- Ion beam analysis data interpretation and consulting
- Elastic Recoil Detection Analysis (ERDA)

## Particle Induced X-ray Emission spectrometry (PIXE)

PIXE is a powerful and relatively simple analytical technique that can be used to identify and quantify trace elements typically ranging from Al to U. Sample irradiation is usually performed by means of 2-3 MeV protons produced by an accelerator (at ANSTO by STAR and 10 MV tandem accelerators). X-ray detection is usually done by energy dispersive semiconductor detectors such as Si(Li) or High Purity Germanium detectors.

## Proton-Induced Gamma-ray Emission spectrometry (PIGE)

When a charged particle (typically protons) approaches the nucleus of a target atom, the coulomb force usually repels it. However when the incident particle has enough energy to overcome the repulsive Coulomb force a charged particle then penetrates through the electrostatic barrier into the nucleus, resulting in interactions with the nuclear forces.

During that process, a number of interactions occur, depending on the energy of the incident particle and the type of target nucleus. Typically, a nuclear reaction will occur, resulting in the emission of high energy x-rays (x-rays emitted from nucleus are for historical reasons called gamma rays) and other nuclear particles.

In PIGE technique, emitted gamma rays are of particular interest as their energies are characteristic of the element and are therefore used to fingerprint elemental composition while yields are used to quantify elemental concentrations.

The detection of the emitted gamma rays is usually done by large volume Ge detectors. PIGE is typically run in conjunction with PIXE and RBS and is used to quantify concentrations of low Z elements such as: Li, F, Na, Mg and Al. Detection limits vary from element to element but are typically between 10 and 100 ppm.

## Rutherford Back-Scattering spectrometry (RBS)

RBS primarily provides information on the profile of concentration versus depth for heavy elements in a light material, e.g. titanium in alumina. Typically, a beam of 2-3 MeV He<sup>+</sup> ions is directed perpendicularly on the sample's surface. As energetic ion penetrates the material it loses energy, mainly in collisions with electrons and only occasionally with nuclei.

When the positively charged He<sup>+</sup> ion comes close to the nucleus of an atom, it will be repelled by positively charged nucleus. The repulsion force increases with the mass of the target atom. For very heavy atoms such as lead or gold, the He<sup>+</sup> ion can be repelled backwards with nearly the same energy as it had before the collision. By measuring the energy spectrum of the recoiled ions, information on the composition of the elements, and their depth within the sample, can be obtained.

## Proton Elastic-Scattering Analysis (PESA)

PESA is similar to RBS but instead of looking at backward angles, the scattered incident ions are measured in forward angles. As the mass separation is rather low in forward angles, only low Z elements like hydrogen can be measured. In this way PESA is ideally suited for measurement of hydrogen concentration in thin samples, like aerosols for example.

## Ion Beam Analysis (IBA) data interpretation and consulting

[IBA](#) is a sensitive technique that can be used to estimate elemental concentrations in solid samples in concentrations from ( $\mu\text{g/g}$ ) to 100% for most elements in the periodic table.

## Elastic Recoil Detection Analysis (ERDA)

ERDA is the complementary technique to RBS, used principally as a method for measuring hydrogen in thin layers, and in the near surface region of materials. In this technique, an incident ion beam is directed at a grazing angle onto the sample's surface. In the ensuing binary collisions with the sample's constituents, recoiling atoms are ejected and are detected at a forward angle.

In ERDA different elemental spectra superimpose on each other, and it is therefore difficult to sort these separate masses so as to provide unambiguous data on the different elemental depth profiles. This however can be done using a somewhat more sophisticated technique called the [Recoil Time-of-Flight spectrometry](#).

Studies of the hydrogen content and profiles of surfaces can be difficult in some cases, due to the pickup of atmospheric moisture by the sample, causing an unwanted hydrogenous background. By using deuterated compounds, the ERDA technique can differentiate between these two isotopes of hydrogen and therefore provide an increased sensitivity.

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