Chemical, Imaging & Mineralogical Facility

University of Exeter, Penryn Campus,

Cornwall, TR10 9FE, UK.

Introduction

The Chemical, Imaging & Mineralogical Facility (CIMF) is located in Cornwall, in Camborne School of Mines (CSM), at the University of Exeter's Penryn Campus. It includes the analysis of chemical elements in inorganic materials, and a wide range of spot and bulk methods available for mineralogical and compositional analysis of natural and synthetic compounds (metals, alloys, ceramics, glass, minerals, rocks, dust, soils, environmental materials, etc.). The facility is available for research, education and limited commercial use and offers a diverse and unique combination of instruments that include;

General

- Sample Preparation – thin rock sections & polished epoxy mounts/blocks.
- Crushing & Grinding – rock/soil sample preparation techniques.
- Optical Microscopy – reflected, transmitted & cold Cathodoluminescence (CL).
- Fluid Inclusion Microscope (Zeiss AX10 scope A1 microscope).

Chemical Analysis

- ICP-MS (Agilent 7700 Series) – Inductively Coupled Plasma Mass Spectrometer, for trace chemical analysis by solution.
- ICP-OES (Agilent 5110 Series) – Inductively Coupled Plasma Optical Emission Spectrometer, for minor & trace chemical analysis by solution.
- LA-ICP-MS (New Wave, NWR 213 ESI) – Laser ablation linked to the above ICP-MS.

Bulk X-ray Analysis

- X-ray Diffraction (Siemens D5000) – XRD, mineral/phase analysis.
- X-ray Fluorescence (Bruker S4 Pioneer) – XRF, elemental analysis.

Electron Beam Microscopy Analysis

- Scanning Electron Microscope with micro-analysis (SEM-EDS) – TESCAN VEGA 3.
- Electron Probe Microanalyser (EPMA) – JEOL JXA-8200.
- Automated Mineralogy by SEM-EDS – QEMSCAN® 4300.

Contacts

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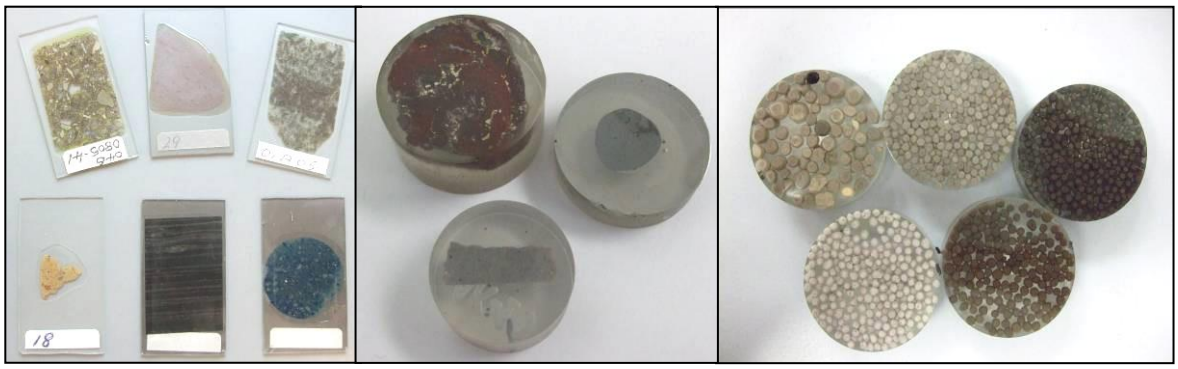
Sample Preparation



Details: Sample preparation of material such as rocks, sediments, particles and ceramics is required for analytical techniques such as optical microscopy and electron beam techniques. This requires the careful preparation of a material into an appropriate form, such as thin sections, polished thin sections, large thin sections (75 by 50 mm) and polished block preparation. Custom preparation of samples such as grain mounts, polished slabs and etching can also be carried out.

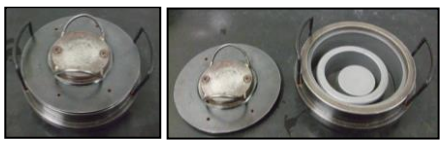
Preparation techniques are water based. Preparation requires cutting, mounting and polishing procedures to achieve a high quality flat finish. The type of preparation is dependant upon the analysis technique and material. If a material is friable or difficult to prepare it may only be possible to produce a polished block.

Preparation type	Details
Thin section (polished)	Suitable for reflected/transmitted optical microscopy and electron beam techniques.
Polished block/mount	Suitable for reflected optical microscopy and electron beam techniques.
Thin section (covered with glass cover slip)	Only suitable for optical microscopy

Crushing & Grinding

Details: Crushing and grinding of material such as rocks and sediments is required for analytical techniques such as XRF, XRD and ICP-MS, where the material needs to be in a powder form. The equipment is **lab-scale** and can handle small rock samples typically the size of a tennis ball or smaller. ***Drying ovens*** (Gallenkamp Hot Box Oven Size 2 & Size 3), ***Jones Riffles*** (bench size), a ***digital balance*** and ***compressed air line hose*** are also available and all processes are done under LEV to contain dust.

Preparation type	Details
Crushing samples	Fritsch Pulverisette: small-scale metal jaw crusher. Approx. sample size that can be crushed = tennis ball to 3mm. (smallest dimensions, long axis may vary).
Grinding samples (by hand)	Mortar & Pestle: ceramic or agate.
Grinding samples (mechanical)	TEMA Mill Unit (Siebtechnick): for ring pots. Approx. size of material that can be loaded per pot = 3mm, mass=20g to 50g.

Ring Mill Type	Contamination (ppm or less typically)	Photograph
<u>Chrome Steel</u> Diameter: 17 cm Weight: 6 Kg	Fe, Cr	
<u>Tungsten Carbide</u> Diameter: 16 cm Weight: 12 Kg	W, Co	
<u>Agate</u> Diameter: 19 cm Weight: 11 Kg	Si	

Optical Microscopy

Nikon Eclipse E600 Pol | **Wild Heerbrugg** | **Cold CL CITL MK5-2** | **Fluid inclusion (Zeiss AX10 scope A1)**

Details: The CIMF is equipped with a range of optical microscopes listed below.

- **Nikon Eclipse E600 Pol microscope**
  This is a reflected and transmitted light microscope with an instant image capture through a Nikon Digital Sight 5MP camera. It is typically used to examine samples prepared as thin sections (both polished & covered) and polished blocks. Data that can be obtained include digital images of areas for texture, fractures or mineral associations, size information (2 mm to 20 microns approx) and determination of opaque mineralogy (metals, ores) and non-opaque/transmitted mineralogy (silicates).

- **Wild Heerbrugg Binocular Microscope**
  This is an optical microscope with optional attached JVC digital camera. It is typically used to examine loose soils and concentrates, or three dimensional surfaces that require low power microscope examination. Data that can be obtained include digital images of areas for texture, fractures or material associations and size information (field of view from 28 mm to 3.5mm approx).

- **Cold Cathodoluminescence Microscope (CITL MK5-2)**
  This is a Nikon Eclipse LV100 optical microscope (fitted with x2, x2.5, x5, x10, x20 objective lenses) with attached Nikon DS-Ri2 digital camera. The CITL MK5-2 electron source is attached to the stage. It is used to examine samples prepared as polished thin sections to study the luminescence characteristics of for example, carbonates, quartz and feldspars for crystal and cement development phases. Data that can be obtained include digital images of areas for texture, cement/crystal growth stages and size information (2 mm to 20 microns approx.).

- **Fluid Inclusion Microscope (Zeiss AX10 Scope A1 microscope)**
  This is an optical microscope with a specialist stage for examining specially prepared samples that contain fluid inclusions. Fluid inclusions are microscopic pockets of liquid or gas trapped in a mineral, which provides information on the physical & chemical conditions of the rock when it formed. The stage is able to cool and heat a sample to examine the behaviour of these inclusions. The microscope has an attached digital camera linked to a computer.

X-ray Fluorescence (Bruker S4 Pioneer)

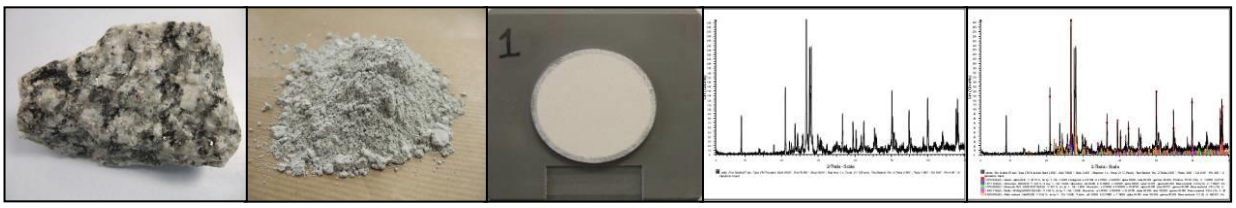
***Details*:** XRF provides bulk chemistry by elemental analysis. Semi-quantitative analysis can be carried out on liquids, loose powders or prepared pressed powder pellets. For fully quantitative analysis pressed powder pellets are used to determine the trace elements, and fused glass beads used for major elements. Sample measurement varies depending on the sample type and program used: typical measurement times vary from 30 minutes to 1 hour per sample. The XRF has an automated 64 sample loader and can operate 24 hours a day. Spectra output are processed using EVAL software.

Analysis type: elemental
Modes: semi-quantitative and quantitative
Detection: % to ppm (element/sample/mode dependant)
Range: F to U
Sample type: solid or liquid
Sample preparation: solids need to be ground to a fine powder (typically less than 50 microns). Pressed powder jackets or fused beads are then prepared.
Data output: tabulated data with quantities of elements present in the sample with error thresholds
Used for: determining elemental presence/concentrations of elements in a sample, such as a rock or soil samples.

X-ray Diffraction (Siemens D5000)

Details: XRD is used to examine bulk mineralogy based on crystallography, with a lower detection limit of ~5%. It cannot examine amorphous material that does not have crystal structure, and will not detect minerals present in a sample below ~5%. Sample measurement typically takes 1 hour with an automatic sample holder that can holder 20 samples and operate 24 hours a day. Output spectra are processed using EVA software.

Analysis type: mineralogical
Modes: qualitative
Detection: approx. 5%
Range: mineral, organic, inorganic databases
Sample type: crystalline solid
Sample preparation: solids need to be ground to a fine powder, typically less than 50 microns. A pressed powder holder (~20g of material required) or a smear sample on a glass slide (for samples 0.5 –1g) is then prepared.
Data output: interpreted spectra with identified minerals/phases present in the sample
Used for: determining the minerals/phases present in a sample, such as a rock or soil sample.

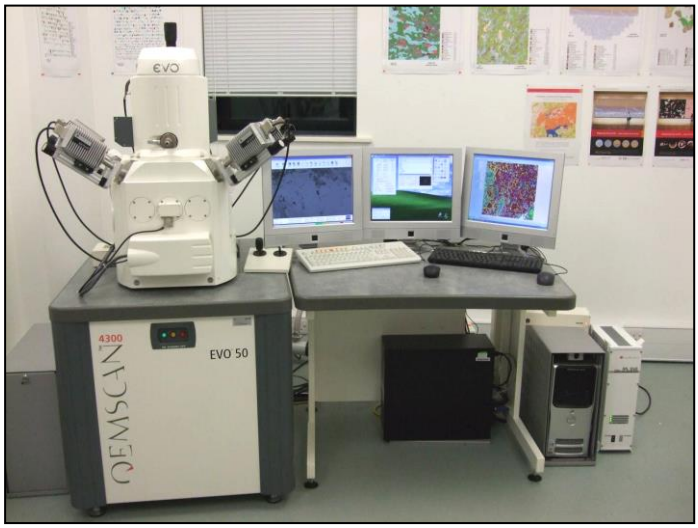


Electron Microprobe (JEOL JXA-8200 Superprobe)

***Details*:** The electron-probe microanalyser (EPMA) is a state-of-the art instrument for the spot analysis and element mapping of minerals and materials; it provides precise chemical compositions of minerals/metals at the micron scale. It consists of an X-ray EDS (Energy Dispersive Spectrometer) detector, 4 wavelength dispersive detectors (2 low count rate detectors for trace elements), a backscatter/secondary electron detector and an optical microscope. The high resolution, low background and automated operation of the instrument enable reliable, quick and precise analysis. Furthermore, it can be used both in spot and a variety of scanning modes to produce accurate chemical analysis, qualitative scans and element distribution maps. The analysis is carried out with an electron beam that is focused on a 1-5 micron spot on the mineral surface. The beam interaction generates X-rays that can be analysed and quantified with respect to standards. The instrument can determine the composition of solid-solution minerals (such as plagioclase feldspar, pyroxene, and titanomagnetite), which can be used to determine crystallization temperatures and pressures, and the incorporation of minor constituents within minerals (such as silver in galena, manganese in ilmenite or gold in pyrite). The instrument can also collect X-ray information over specified areas on a sample surface to produce maps of element distributions. The instrument enables users to work on four thin sections or polished blocks at a time and can operate unattended over nights and weekends. For a routine analytical setup, users are usually able to collect 50-80 mineral analyses during a full day.

Analysis type: elemental
Vacuum modes: high (7 x 10^{-4} Pa)
Accelerating voltage: 0.5 to 30 KV
Electron Gun: tungsten filament
Imaging modes: SEI, BEI
Measurement modes: qualitative**,** quantitative**,** line scan**,** mapping**,** particle analyser
Detectable elements: ^{5}B to ^{92}U. X-ray analysis (1% approx). Wavelength dispersive analysis (% to ppm)
Sample type: solid (polished block/polished thin section)
Sample preparation: samples need to be in polished thin section form or rock fragments, soils, powders and concentrates mounted in polished resin blocks (25 or 30 mm diameter).
Data output: Semi-quantitative/quantitative chemical data for each analysis point (tabulated), digital greyscale photomicrographs, qualitative chemical data and element maps.
Used for: Determining elemental presence/concentrations of elements in a point, such as a rock, soil metal or ceramic samples. Inferring the mineralogy of samples from spot X-ray chemical analysis to validate optical microscopy. Imaging of a sample surface for texture, fractures or surface features.

QEMSCAN® (4300, EVO 50)



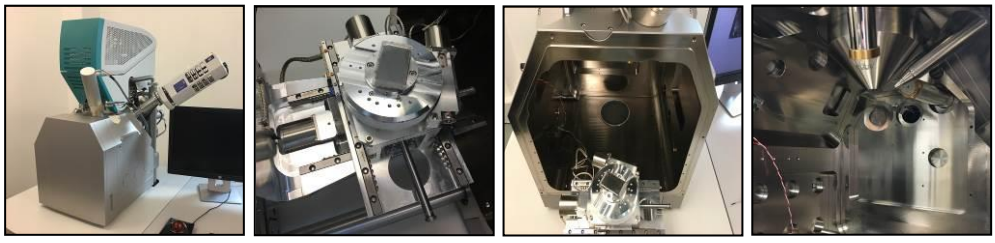
Details: The QEMSCAN® is used for the Quantitative Evaluation of Minerals (QEM) or inorganic phases using a Scanning Electron Microscope (SEM). It is based on a Zeiss EVO 50 series SEM and consists of four light element Bruker SDD (Silicon Drift Droplet) Energy Dispersive X-ray Spectrometers (EDS) and an electron backscatter detector. The QEMSCAN® is a fully automated computer controlled instrument and can run 24 hours a day. The instrument uses an automated image analysis system that combines both backscatter (BSE) and energy-dispersive X-ray signals to identify minerals and inorganic chemical compounds. Analysis points return X-ray spectra that are compared with a large database, allowing the assignment of each analysis point to a specific mineralogy and chemistry. Each analysis point can achieve approximately 200,000 counts per second using digital pulse processors and takes approximately 5 to 10 milliseconds to measure, with a feature detection limit of 0.2 microns. Output data is processed using proprietary iDiscover software by an experienced operator.

Analysis type: mineralogical
Modes: high vacuum
SEM: Zeiss EVO® 50
Detection: ^{6}C to ^{92}U. X-ray analysis (1% approx. in manual mode). Feature detection to 0.2 microns.
Sample type: solid mineral or inorganic (polished block/polished thin section)
Sample preparation: samples typically need to be in thin section form (47 by 25 mm) or rock fragments, soils, powders and concentrates mounted in polished resin blocks (30 mm or 25 mm diameter). It is possible to examine samples mounted on filter papers depending on the filter paper type, large thin sections (75 by 50 mm) and minimal sample size via smear mounts.
Measurement modes: 5 modes (customisable); PMA (particle mineral analysis), BMA (bulk mineral analysis, line scan), TMS (trace mineral search), SMS (specific mineral search), Fieldscan (x-ray analysis for textural information). Measurement time depends on the mode and level of detail required. 4000 particles mapped to a reasonable detail using PMA mode typically takes 4 hours, a rock fragment in a thin section scanned at 10 microns in fieldscan mode can take up to 10 hours.
Data output: tabulated data such as modal mineralogy (percent), mineral associations, liberation, false colour images/maps of sample areas or particles
Used for: examining the mineralogy and texture of a sample, and or liberation studies linked to mineral processing.

SEM-EDS (TESCAN VEGA3 GMU)

Details: The SEM (scanning electron microscope) uses an electron beam to examine a sample. The instrument consists of an X-ray EDS (Energy Dispersive Spectrometer) detector and a backscatter/secondary electron detector that are used in combination to evaluate a sample. The backscatter /secondary detector records electrons emitted from a sample for visual detail and the X-ray EDS detector records X-rays to enable qualitative and semi-quantitative chemical analysis. The instrument can be operated in either low vacuum or high vacuum mode.

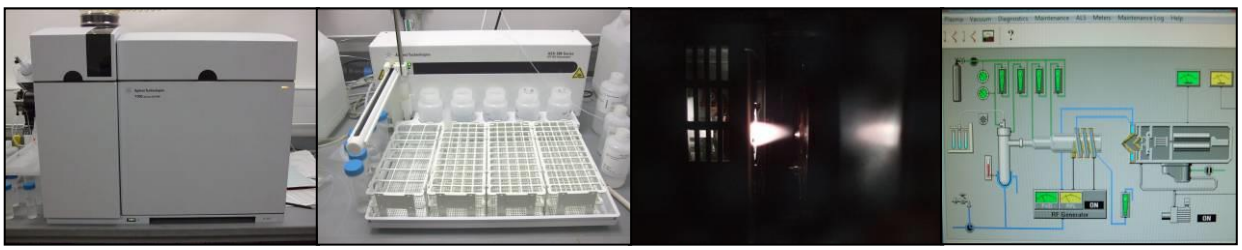
Analysis type: imaging, with chemical analysis for inorganic material.
Vacuum modes: low vacuum: 3 – 500 Pa^5 (optionally: 3 – 2000 Pa^5)
high vacuum: < 9×10^{-3} Pa^2
Accelerating voltage: 0.2 KV to 30 KV
Electron gun: LaB_6 or tungsten filament
Imaging modes: SE, BSE
Analysis Functions: Oxford EDS system (XMax 80mm EDS), Aztec software (version 3.3 SP1).
Detectable elements: ^6C to ^{92}U. X-ray analysis detection to 1% (approx.).
Specimen stage: Compucentric. X= 130mm, Y= 130mm, Z= 100mm, T= -80 to +90, R= 360.
Chamber size: 340mm (w) x 315mm (d). Max specimen height: 106mm with rotation, 147mm without rotation stage.
Sample preparation: the low vacuum facility allows objects to be placed into the chamber with little or no preparation. High vacuum mode generally requires a sample to be carbon coated. Samples that can be examined include rock fragments, powders or soils mounted in resin blocks or on a stub, polished thin-sections, smear mounts, filter papers or any organic or inorganic material that is stable under vacuum.
Data output: greyscale photomicrographs (images), qualitative, quantitative chemical data.
Used for: imaging of a sample surface for texture, fractures or surface features. Determining elemental presence/concentrations in a sample, such as a rock or soil samples. Inferring the mineralogy of samples from spot X-ray chemical analysis to validate optical microscopy. Elemental maps of samples.



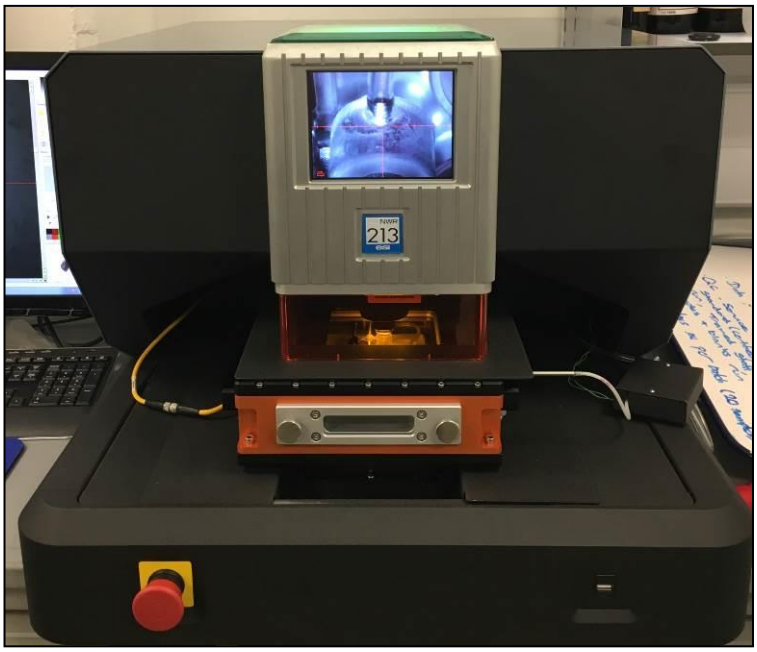
Inductively Coupled Plasma – Mass Spectrometer (Agilent 7700 Series)

Details: ICP-MS can measure a full suite of elements in a single multi-element acquisition, accepts any sample type in solution and can provide isotopic information. The 7700 has a Quadruple mass spectrometer and is able to work in both He (collision) and H_2 (reaction) mode. Sample measurement time varies depending upon the number of elements required. The instrument has an automated sample holder that can hold up to 240 samples and operate independently.

Analysis type: chemical
Modes: quantitative
Detection: ppm, ppb, ppt, Li to U.
Sample type: solutions (liquid)
Sample preparation: samples are prepared into solution by block digestion using a selection of acids, depending upon material type.
Data output: tabulated data with quantities of elements present in the sample.
Used for: determining the presence/concentrations of elements in a solution, which could be from a rock, soil or water sample.



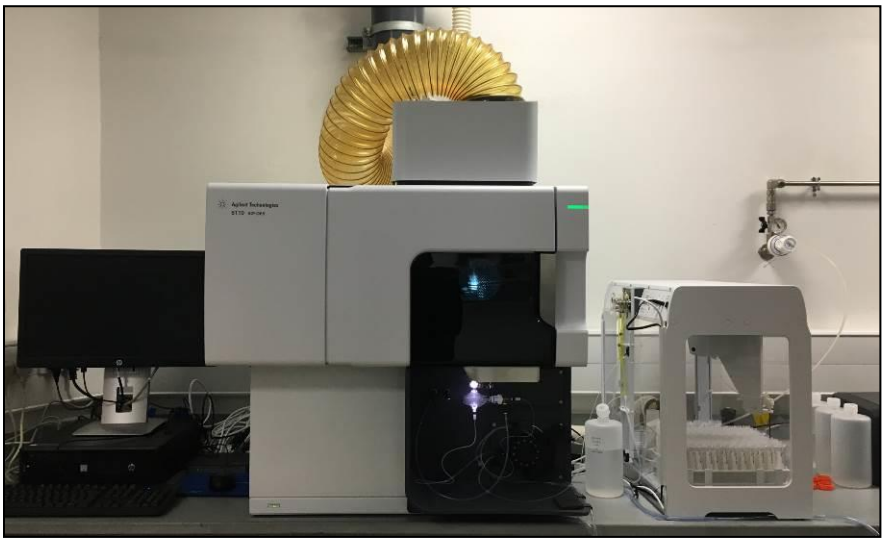
LA-ICP-MS (New Wave, NWR 213 ESI)



Details: Laser ablation-ICP-MS is designed to analyse elements by ablating a spot or line from a solid sample, the volatile material is then analysed by ICP-MS. Therefore the laser is a separate unit that is coupled to the ICP-MS. It can measure multiple elements in a single acquisition. Details of the laser and ICP-MS are below. The laser is couple to an Agilent ICP-MS 7700 that has a Quadruple mass spectrometer.

Analysis type: chemical.
Modes: quantitative/qualitative
Laser: Nd:YAG 213 nm (infrared). 1 to 20Hz repetition rate.
Spot size: 4µm to 250µm.
Chamber: 100mm by 100mm
Detection: ppm, ppb, ppt, Li to U.
Sample type: solid material (rocks, minerals, metals, ceramics).
Sample preparation: samples must be prepared into thin sections (47 x 25 mm approx.) or blocks (25mm diameter 13mm thick approx.).
Data output: tabulated data with quantities of elements present in the sample.
Used for: determining the presence/concentrations of elements in a sample (spot or line analysis).

Inductively Coupled Plasma – Optical Emission Spectroscopy (Agilent 5110 Series)



Details: ICP-OES can measure a full suite of elements simultaneously and accepts samples that have been prepared into a solution. Analyte concentrations are derived from characteristic energy emission of the elements which are collected via a CCD detector in the visible and UV spectrum (wavelengths from 785 to 167 nm). Sample measurement time is affected by the measurement settings that are linked to the requirements of the analysis but is typically about 150 seconds per sample. The instrument has an automated sample holder that can hold up to 240 samples.

Analysis type: chemical.
Modes: quantitative
Detection: ppm, ppb, Li to U.
Sample type: solutions (liquid)
Sample preparation: samples are prepared into solution either by block digestion or basic dissolution, using a selection of acids, depending upon material type.
Data output: tabulated data with quantities of elements present in the sample.
Used for: determining the presence/concentrations of elements in a solution, which could be from a rock, soil or water sample.