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Laser Ablation Inductively Coupled Plasma Mass Spectrometry

(LA-ICP-MS)

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Introduction

Inductively coupled plasma mass spectrometry (ICP-MS) is undoubtedly the fastest-growing trace element technique available today. Since its commercialization in 1983, approximately 5000 systems have been installed worldwide, carrying out many varied and diverse applications. The most common ones, which represent approximately 80% of the ICP-MS analyses being carried out today, include environmental, geological, semiconductor, biomedical, and nuclear application fields.

There is no question that the major reason for its unparalleled growth is its ability to carry out rapid multielement determinations at the ultra trace level. Even though it can broadly determine the same suite of elements as other atomic spectroscopical techniques, such as flame atomic absorption (FAA), electrothermal atomization (ETA), and inductively coupled plasma optical emission spectroscopy (ICPOES), ICP-MS has clear advantages in its multielement characteristics, speed of analysis, detection limits, and isotopic capability. Figure 1.1 shows approximate detection limits of all the elements that can be detected by ICPMS, together with their isotopic abundance.

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What is ICP-MS and how does it work?

The sample, which usually must be in a liquid form, is pumped at 1 mL/min, usually with a peristaltic pump into a nebulizer, where it is converted into a fine aerosol with argon gas at about 1 L/min. The fine droplets of the aerosol, which represent only 1–2% of the sample, are separated from larger droplets by means of a spray chamber. The fine aerosol then emerges from the exit tube of the spray chamber and is transported into the plasma torch via a sample injector. Once the ions are produced in the plasma, they are directed into the mass spectrometer via the interface region, which is maintained at a vacuum of 1–2 Torr with a mechanical roughing pump. This interface region consists of two metallic cones (usually nickel), called the sampler and a skimmer cone, each with a small orifice (0.6–1.2 mm) to allow the ions to pass through to the ion optics, where they are guided into the mass separation device. The interface region is one of the most critical areas of an ICP mass spectrometer because the ions must be transported efficiently and with electrical integrity from the plasma, which is at atmospheric pressure (760 Torr) to the mass spectrometer analyzer region at approximately 106 Torr. Once the ions have been successfully extracted from the interface region, they are directed into the main vacuum chamber by a series of electrostatic lens, called ion optics. The operating vacuum in this region is maintained at about 103 Torr with a turbomolecular pump. There are many different designs of the ion optical region, but they serve the same function, which is to electrostatically focus the ion beam toward the mass separation device, while stopping photons, particulates, and neutral species from reaching the detector. The ion beam containing all the analytes and matrix ions exits the ion optics and now passes into the heart of the mass spectrometer—the mass separation device, which is kept at an operating vacuum of approximately 106 Torr with a second turbomolecular pump. There are many different mass separation devices, all with their strengths and weaknesses. Four of the most common types are discussed in this book—quadrupole, magnetic sector, time of flight, and collision/reaction cell technology—but they basically serve the same purpose, which is to allow analyte ions of a particular mass-to-charge ratio through to the detector and to filter out all the nonanalyte, interfering, and matrix ions. Depending on the design of the mass spectrometer, this is either a scanning process, where the ions arrive at the detector in a sequentially manner, or a simultaneous process, where the ions are either sampled or detected at the same time. The final process is to convert the ions into an electrical signal with an ion detector. The most common design used today is called a discrete dynode detector, which contain a series of metal dynodes along the length of the detector. In this design, when the ions emerge from the mass filter, they impinge on the first dynode and are converted into electrons. As the elec-trons are attracted to the next dynode, electron ultiplication takes place, which results in a very high steam of electrons emerging from the final dynode. This electronic signal is then processed by the data handling system in the conventional way and then converted into analyte concentration using ICP-MS calibration standards. Most detection systems used can handle up to eight orders of dynamic range, which means that they can be used to analyze samples from ppt levels, up to a few hundred parts per million (ppm). (Thomas, 2004)

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How does ICP-MS compare to other analytical methods?

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What is the difference between ICP-MS and laser ablation?

- A focused laser beam directed onto the surface of a mineral grain is used to excavate, or “ablate,” a small volume of sample.
- The ablated material, in the form of micrometer-size fragments and condensed droplets, is transported by a carrier gas into the induction port of an ICP where the solids are melted, dissociated, and ionized.
- The ions are then focused into a quadrupole mass spectrometer where detection of a broad range of elemental masses occurs.
- The technique offers a wide dynamic range with trace and ultratrace detection limits coupled with simultaneous major-element determinations.
- However, the introduction of solid sample material to the instrument as opposed to the usual solution creates a new set of complexities.
    - The mechanical, chemical, and physical factors involved in laser ablation and transport of the sample to the instrument result in a variety of fractionation effects (Ludden and others, 1995).
    - Elemental fractionation occurs primarily because of secondary heating of the sample crater following the laser shot and thus, consequently, is of particular concern in infrared laser determinations.
    - Elements more volatile than the host material can be volatilized from the area surrounding the crater and carried to the plasma, resulting in erroneously high element concentrations. For an element such as mercury this can be as much as 30-percent error in a poorly designed experiment. (Taylor et al. 2010)

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How does LA-ICPMS work?

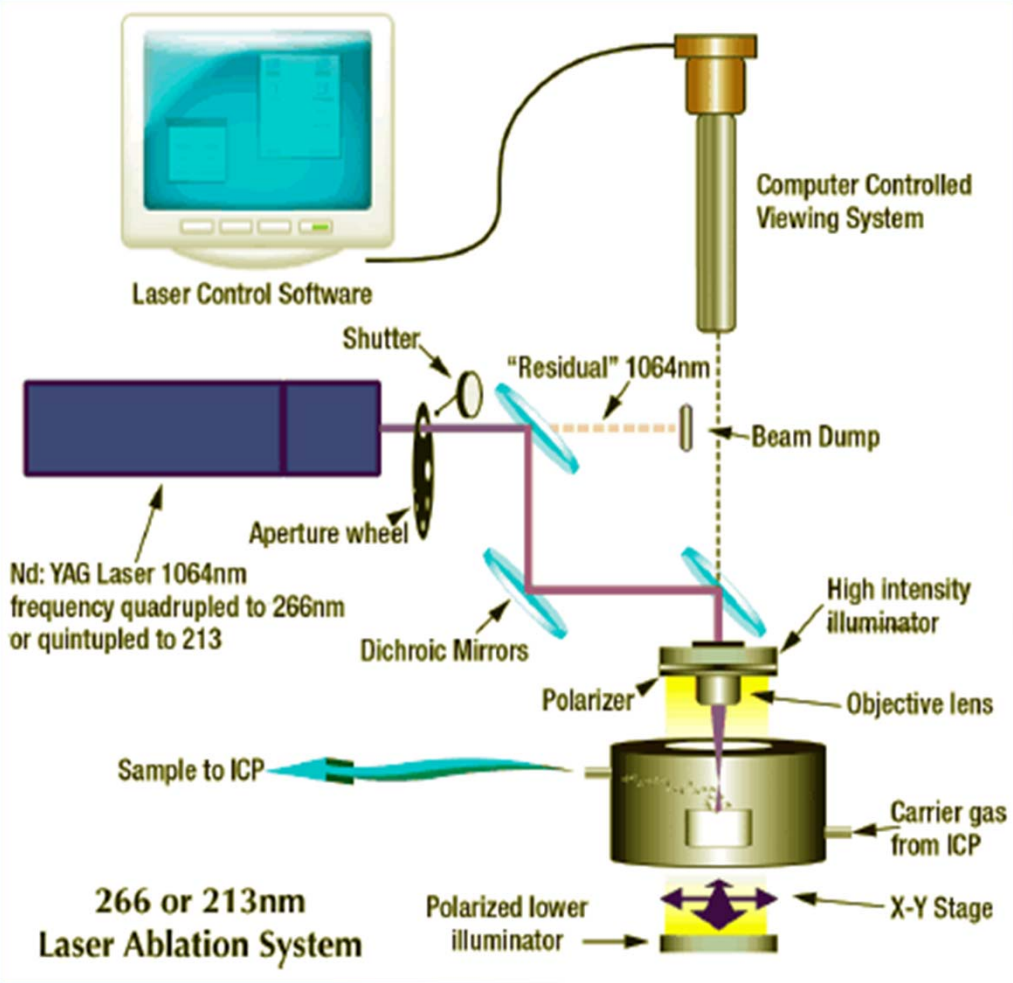

Diagram of a laser ablation system

http://www.gasesmag.com/articles.php?pid=36

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LA-ICP-MS system

Best technique for detecting trace amounts of PGE to a few tens of ppb.
Laser beam is 40µm
Can ablate lines, spots, shapes, even words

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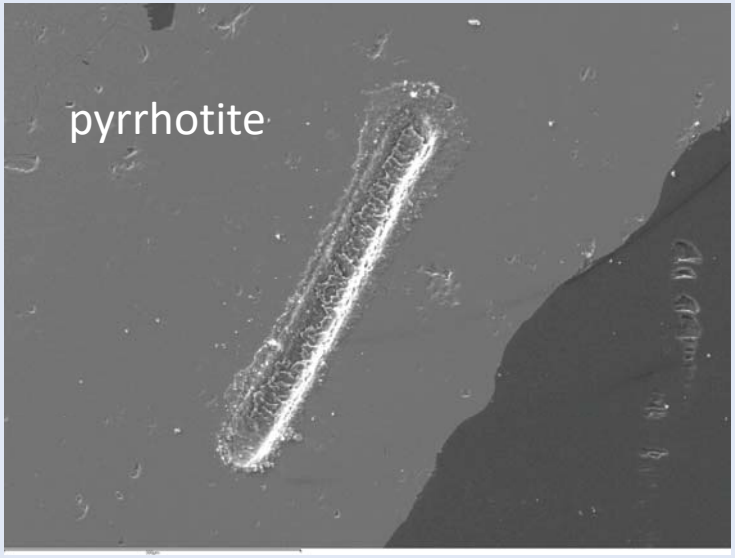
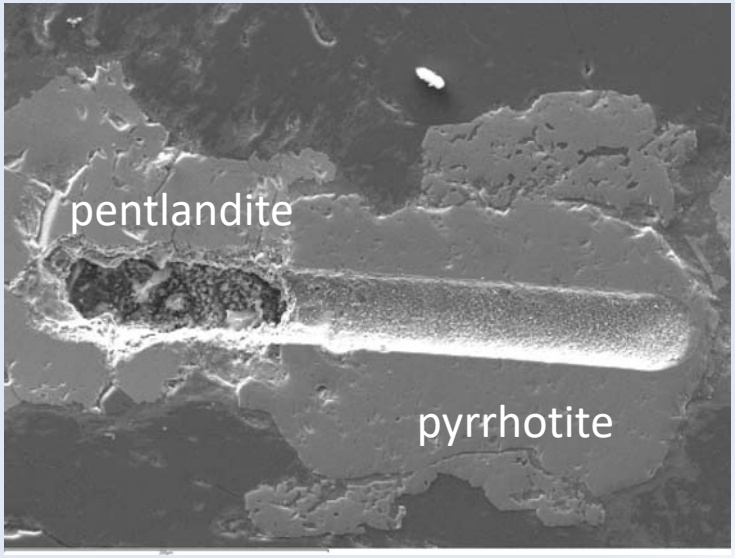
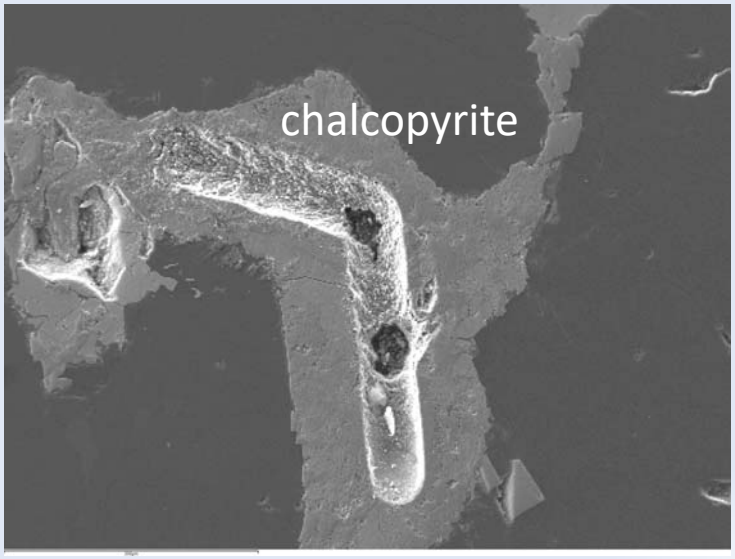
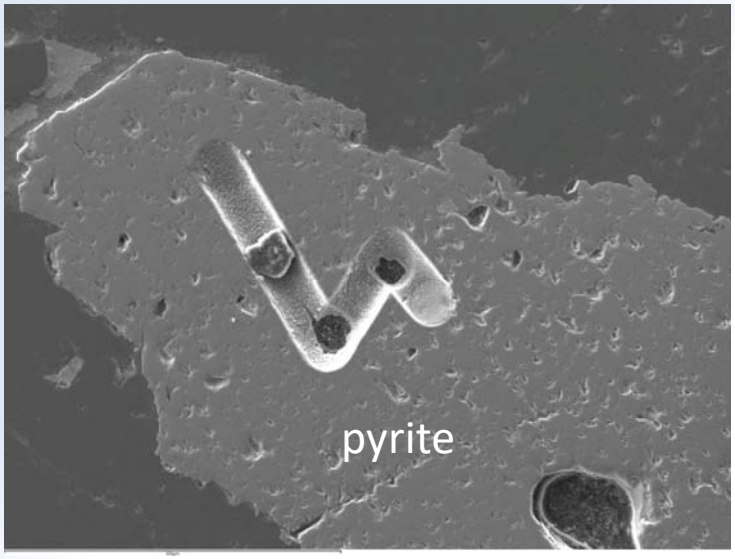
Ablation Pits









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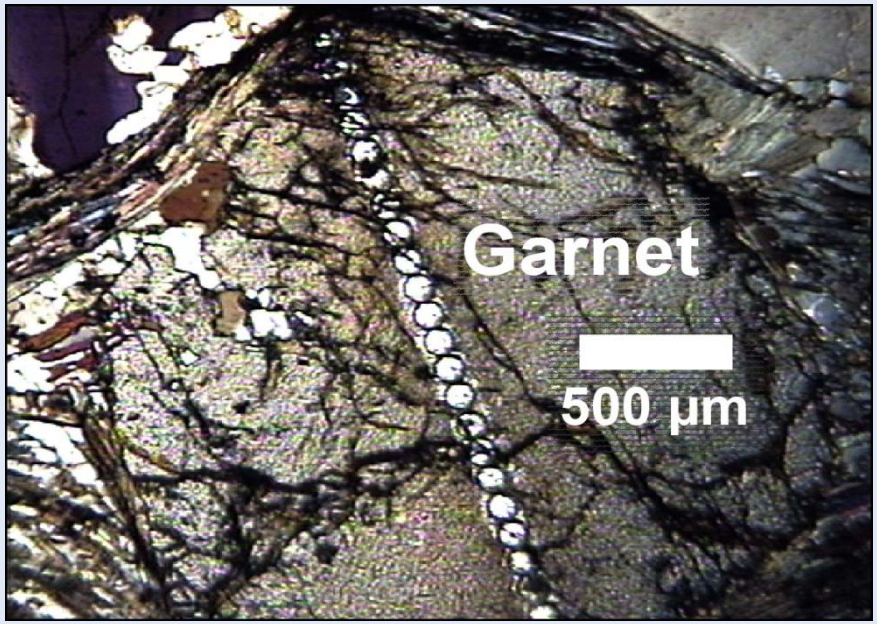
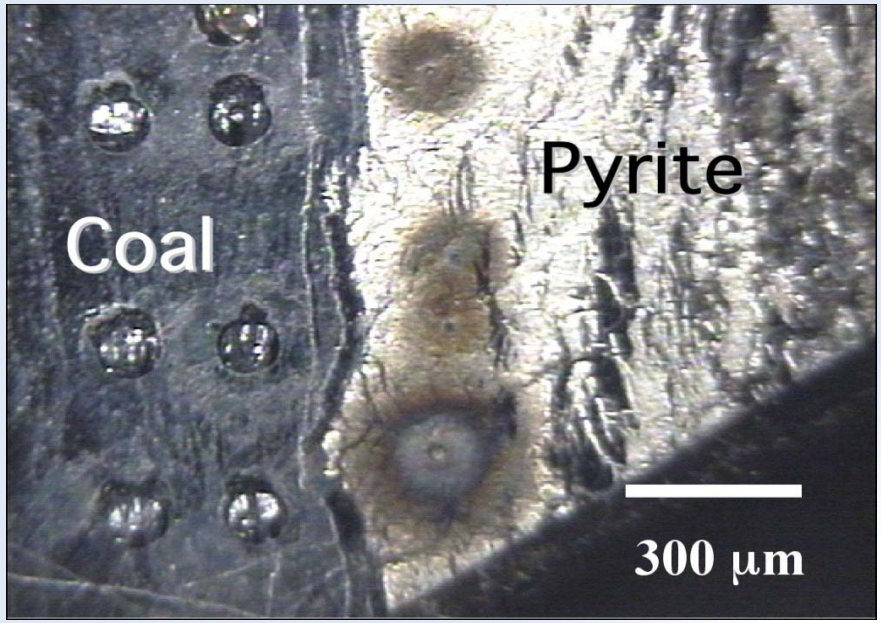
LA-ICP-MS system





The photomicrographs above show the detail of craters ablated by the laser in the solid materials. The spot sizes range from 100 microns in the garnet (above left) and the coal (above right) and 10 microns in the pyrite (above right).

http://crustal.usgs.gov/laboratories/icpms/laser_ablation.html

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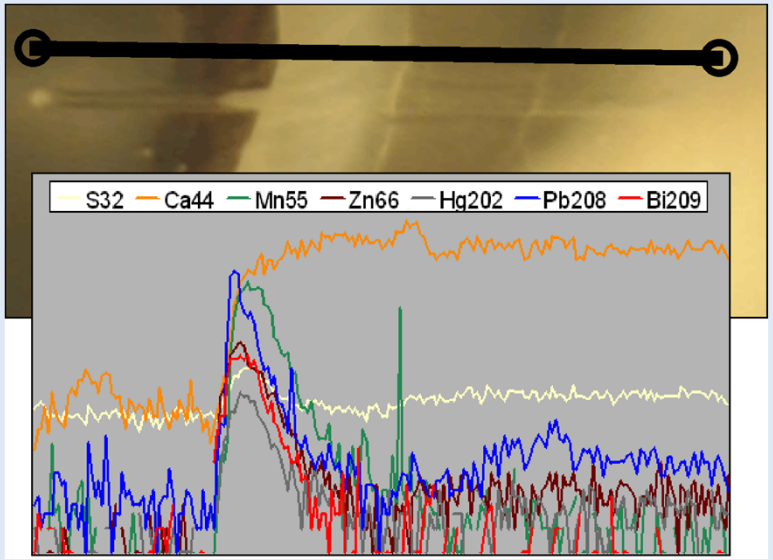
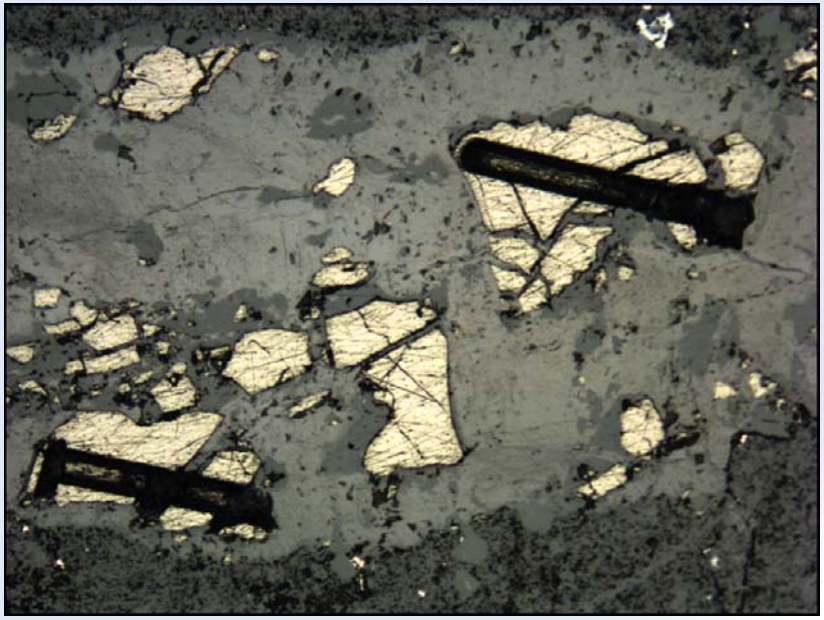
LA-ICP-MS system



Analysis of moose teeth: LA-ICP-MS line scan through glue, dark material, enamel and dentine. The graph is raw ICP-MS signal (cps). Note the enrichment of Mn, Zn and Pb in this dark material. The Pb signal in this analysis is higher than most others of the dark material.



The photomicrograph above shows LA line scans ablated in bitumen (50 micron diameter lines)

http://crustal.usgs.gov/laboratories/icpms/laser_ablation.html

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What are the advantages of LA-ICP-MS?

- LA offers a method with very good selectivity and sensitivity:
- Reduced sample preparation time - Dissolution methods involve sampling a portion of the solid material under investigation, placing the sample aliquot (usually milligrams) into a digestion vessel, adding concentrated acid(s) and finally digesting the material with use of a controlled heating device over a specified period of time (usually several hours or more).
- The reduced sample preparation time removes some of the contamination issues, including contributions from the sample container, from the added solvents (acids and water), and from the atmosphere.
- Reduces the chance of sample and analyte (volatile components) loss and reduces serious exposure-related hazards that must be considered when heating concentrated acid solutions
- Smaller relative sample size - generally in the sub-microgram range for most ablation methods versus milligrams of material (or more) needed for dissolution methods
- Reduced sample sizes are especially beneficial to certain applications (i.e. forensics) where there are often limited amounts of sample.
- As a result to the small amount of sample consumed, laser ablation is considered a nondestructive technique (or virtually nondestructive) and hence classified as a microchemical analysis. (Almirall et al. 2012)
- In addition, the smaller amount of mass entering the ICP-MS for laser ablation typically translates into higher detection limits.

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What are the disadvantages of LA-ICP-MS?

- First of all, since the consumption of sample is much less in comparison to dissolution methodologies, the sample is (or can be) less representative of the bulk (whereas dissolution/digestion procedures are considered bulk analyses).
    - Multiple sampling locations can increase sample representation and thus enhance characterisation.
- In terms of quantitative analysis, quantification of the ablated mass is often difficult where matrix matched standard reference materials are not available.
- The reason matrix match standards are important is because accurate quantification by laser ablation (ICP-MS) is directly correlated to the ablation rate (the amount of mass ablated per laser pulse), which is inherent to the respective sample matrix. Thus, if the sample set under investigation is of different composition than the standard being used for quantitative analysis, the laser to sample interaction (and ultimately sampling) is different and which makes the association inaccurate. In other words, even with similar compositions some assumptions must be made when performing quantitative analysis by LA-ICP-MS.
- However, despite the lack of matrix-matched standards, some applications have utilized the NIST series glass standards for quantification and successful results have been obtained in terms of accuracy nevertheless this protocol is not recommended.
- Another disadvantage and probably the most studied variable related to laser ablation is elemental fractionation, which occurs (or is defined as when) the ablated mass is different in composition from the bulk sample. Fractionation can be intrinsic (matrix related) and/or it can occur as a function of the ablation process (dependency on laser irradiance, wavelength, pulse width, and pulse duration); fractionation can even be a product of ablation transport (in relation to carrier gas and the ablation chamber/tubing) and/or it can occur within the inductively coupled plasma itself. Research has shown that utilizing a higher laser irradiance and shorter pulse durations significantly reduces fractionation, these variables aid in producing smaller particle size distributions that are more readily transported and efficiently atomized/ionized in the inductively coupled plasma. (Almirall et al. 2012)

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What are the application of LA-ICP-MS in the real word?

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How can LA-ICP-MS aid exploration?

Technical services based on LA-ICPMS

- Gold mineralogy
- Refractory Au versus inclusions, plus size and chemistry of inclusions.
- Comprehensive gold deportment
- Determines gold mineralogy and how much of the gold is contained in each mineral.
- Elemental concentrations in minerals from rocks ores, concentrates or tailings
- Most elements in the periodic table can be analysed.
- U-Pb geochronology
- Zircon, monazite, sphene, apatite and uraninite.
- Mineral mapping and sulfide ore paragenesis
- Mineral zonation and growth history.
- Mineral chemical vectoring and fingerprinting by LA-ICP-MS
- Pyrite, epidote, chlorite and others.

Generic pricing?

http://www.utas.edu.au/__data/assets/pdf_file/0005/404582/CODES_Technology-Brochure_website.pdf

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What are the costs and processing times?

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Conclusions

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Further reading

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What is ICP-MS?

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What is LA-ICPMS?

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a

Laser ablation is the process of removing material from a solid (or occasionally liquid) surface by irradiating it with a laser beam. At low laser flux, the material is heated by the absorbed laser energy and evaporates or sublimates. At high laser flux, the material is typically converted to a plasma.

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Laser Ablation Mass Spectrometry

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LA-ICPMS system

The two types of gold, as well as native silver, are easily distinguished based upon LA–ICP–MS geochemical studies of free gold separates recovered from the mill (figs. 9A–I). Laser traverses across 0.5- to 1.0-mm-sized grains recovered from heavy concentrates, represented as time versus elemental intensity plots, clearly indicate the presence of nearly pure gold grains with a high ratio of gold to silver (figs. 9A, D–F). Other grains that appear silvery-white on polished surfaces show lower gold-to-silver ratios and consistently elevated mercury, demonstrating that the silvery white gold is actually a gold-silver-mercury amalgam (figs. 9B and C). A few traverses suggest that some of the gold grains may also incorporate significant copper (fig. 9G). Silver grains either contain irregular inclusions of gold or are relatively pure (figs. 9H and I). The silver quickly tarnishes to a dirty, bluish-gray color, enabling visual identification in slides of the mill separates.

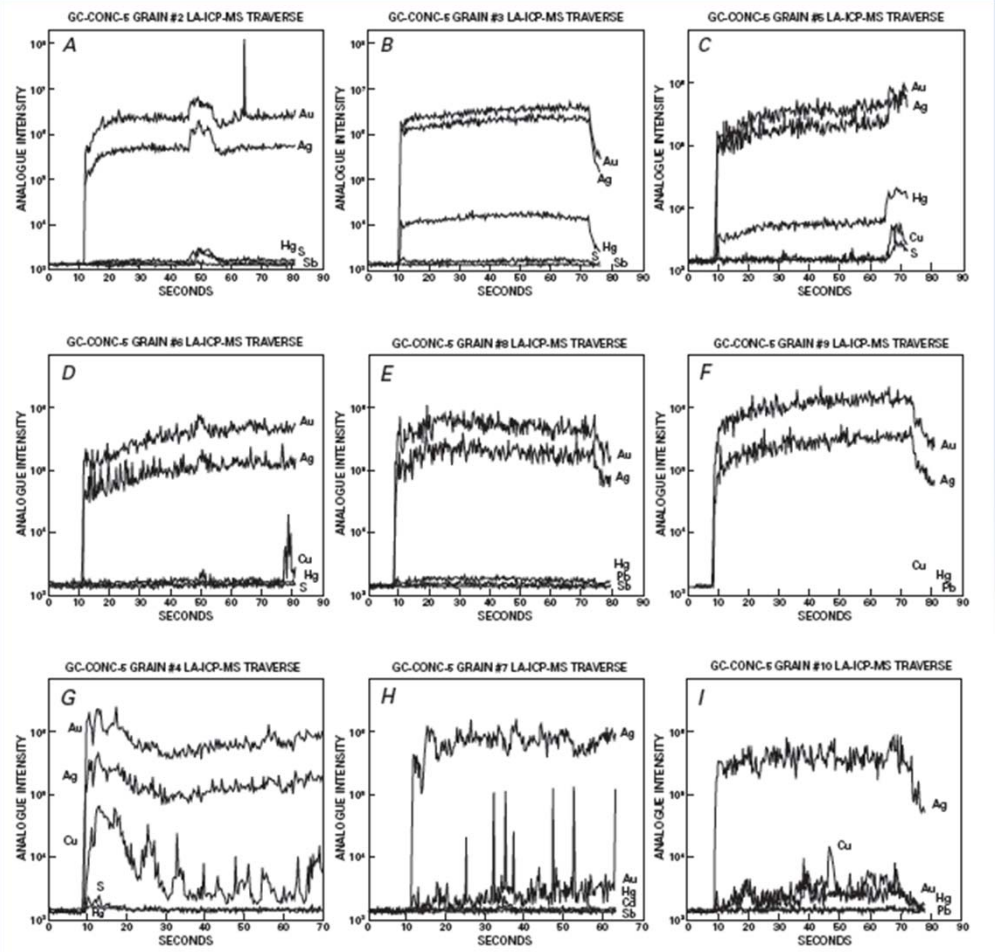

Figure 9. LA–ICP–MS traverses across heavy concentrate grains recovered from the Greens Creek mill. All grains analyzed are from the coarsest fraction of sample GC–CONC–5. Each profile was obtained by operating the laser in continuous mode across a traverse of approximately 100 micrometers with the ICP–MS detectors set to record the relative abundances, measured as the $\log_{10}$ of analogue intensity, of five selected elements. Each plot exhibits the relative abundances of the elements selected and shows the variation in relative concentration with time (distance along the traverse). Note that plots *A* and *B* have a greater y-axis range than plots *C–I*. Plots a and b exhibit the two types of gold identified at Greens Creek: yellow gold of high fineness and silver-white electrum composed of an amalgam of gold, silver, and mercury. Plot *C* is an example of electrum, plots *D–F* are analyses of yellow gold grains, plot g represents a possible copper-rich variant of yellow gold, and plots h and i are examples of nearly pure native silver grains. (*A*) grain #2, Au and Ag only, high Au/Ag; (*B*) grain #3, Au, Ag, and Hg, lower Au/Ag; (*C*) grain #5, Au, Ag, and Hg, lower Au/Ag; (*D*) grain #6, Au and Ag, high Au/Ag; (*E*) grain #8, Au and Ag, high Au/Ag; (*F*) grain #9, Au and Ag, high Au/Ag; (*G*) grain #4, Au, Ag, and Cu, high Au/Ag; (*H*) grain #7, nearly pure Ag; and (*I*) grain #10, nearly pure Ag.

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LA-ICP-MS system

Method	Capital cost $ (approx.)	Multielement	Precision	Sample type ($US)	Cost per	comments sample
Colorimetry	8000	No	Poor	Solution	2–10	Good for field use and W, Mo
Atomic absorption spectrophotometry	60,000	No	Good	Solution	1–5	Cheap and precise
X-ray fluorescence	200,000	Yes	Good	Solid	20	Good for refractory elements
ICP–ES	150,000	Yes	Good	Solution	10	Good for transition metals
ICP–MS	150,000–1000,000	Yes	Good	Solution	10	Good for heavy metals. High resolution instruments very low detection limits

Other geochemical methods used in exploration (Moon et al. 2006)

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LA-ICP-MS system

The differences between the methods shown are cost, the detection limits of analysis, speed of analysis, and the need to take material into solution. Most general analysis in developed countries is carried out by inductively coupled plasma emission spectrometry (ICP–ES), often in combination with inductively coupled plasma mass spectrometry (ICP–MS), or X-ray fluorescence (XRF). All three methods require highly sophisticated laboratories, pure chemicals, continuous, non-fluctuating power supplies, and readily available service personnel, features not always present in developing countries. In less sophisticated environments, high quality analysis can be provided by atomic absorption spectrophotometry (AAS), which was the most commonly used method in developed countries until about 1980. Another method which is widely used in industry is neutron activation analysis (NAA) but its use is restricted to countries with cheap nuclear reactor time, mainly Canada. Precious metals (gold and platinum group elements) have been extremely difficult to determine accurately at background levels. The boom in precious metal exploration has, however, changed this and commercial laboratories are able to offer inexpensive gold analysis at geochemical levels (5 ppb to 1 ppm) using solvent extraction and AAS, ICP–ES or alternatively NAA on solid samples. For evaluation the method of fire assay is still without equal: in this the precious metals are extracted into a small button which is then separated from the slag and determined by AAS, ICP–ES, or ICP–MS. The analysis of precious metals is different from most major elements and base metals in that large subsamples are preferred to overcome the occurrence of gold as discrete grains. (Moon et al. 2006)

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LA-ICP-MS system

The LA-ICPMS laboratory is used for direct in-situ elemental and isotope-ratio analysis of solid samples. We utilise a custom-designed/built deep-UV 193 nm excimer laser-ablation system coupled to an Agilent 7500ce/cs quadrupole ICPMS (or a GV Instruments IsoProbe MC-ICPMS). The laser-ablation system was designed in close collaboration with Mike Shelley (Laurin Technic P/L, Canberra, Australia) and Resonetics LLC (Nashua, NH, USA) and is the prototype of the system now marketed as RESOlution M-50 (http://www.resonetics.com/la/). At its core, it features a Laurin two-volume laser-ablation cell characterized by both fast signal washout and uniform signal characteristics anywhere in the cell.

The system is extremely versatile, fully computer-controlled and easy to use even for beginners, since all is controlled by an intuitive image-directed software (Geostar). The large primary excimer laser beam in conjunction with aperture-imaging optics and a long working distance lens allows the use of a large range of round spot sizes from ~7 – 300 µm. Moreover, this includes a rotating adjustable rectangular slit that is especially useful for layered samples where the highest-spatial resolution along a growth axis has to be maintained without loss of element detection capability. X Y stage travel is 50 x 50 mm.

We also have a unique cryo-sample holder (developed in collaboration with Laurin Technic) for the analysis of frozen samples, which is currently used for elemental analysis of frozen Greenland ice core samples at unprecedented spatial resolution.

http://www.rhul.ac.uk/earthsciences/labs/laicpms.aspx

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LA-ICP-MS system

The first questions a user is likely to have when considering the use of LAICP-MS are often concerned with the sort of data likely to be obtained, how long the analysis will take, sample's suitability and so on. In other words, why use this technique?

The advantages of using LA-ICP-MS for the analysis of solids are fairly clear. Samples are analysed in the solid state and so there is no need for lengthy digestion and dissolution steps. Most inorganic solids can be analysed, along with some organic solids such as wood and body tissues. Spatial information can be obtained, at resolutions varying between five and a few hundred microns. The technique is extremely sensitive, providing low level trace element data for almost all elements from Li to U, in a single analysis which could take just a minute or two. Isotope ratios may be determined. Samples, although almost always prepared, can on occasion be analysed without any preparative steps. The technique is less prone to interferences than many other solid sampling techniques such as ion probe microanalysis or electron microprobe analysis. The disadvantages of the technique are not so immediately obvious except that it is sample destructive on a microscopic scale. It is not a primary analytical technique and requires the use of an internal standard to correct for variation in ablation yield between standards and samples. This internal standard must be measured beforehand by a technique such as electron microprobe analysis. With modern systems, phenomena such as laser induced elemental fractionation are less of a problem than in the past, but remain a challenge. The physics and chemistry of laser-sample interaction are poorly understood. Finally, as much an advantage as a disadvantage, the technique is young and still in rapid development. (Jeffries, 2004)

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LA-ICP-MS system

One of the largest volume uses for ICP-MS is in the medical and forensic field, specifically, toxicology. A physician may order a metal assay for a number of reason
Depending on the specific parameters unique to each patient's diagnostic plan, samples collected for analysis can range from whole blood, urine, plasma, serum,
field. Such applications include water testing for municipalities or private individuals all the way to soil, water and other material analysis for industrial purposes.
In recent years, industrial and biological monitoring has presented another major need for metal analysis via ICP-MS. Individuals working in plants where exposu
have their blood or urine analyzed for metal toxicity on a regular basis. This monitoring has become a mandatory practice implemented by OSHA, in an effort to
rotating employees from a high exposure position to a low exposure position).
Regardless of the sample type, blood, water, etc., it is important that it be free of clots or other particulate matter, as even the smallest clot can disrupt sample f
e.g. sodium chloride in sea water, can eventually lead to blockages as some of the ions reunite after leaving the torch and build up around the orifice of the skim
suspected, though at a cost to detection limits.
This technique is also widely used in the field of radiometric dating, in which it is used to analyze relative abundance of different isotopes. ICP-MS is more suitabl
with high ionization energy such as osmium and tungsten can be easily ionized.
In the field of flow cytometry, a new technique uses ICP-MS to replace the traditional fluorochromes. Briefly, instead of labelling antibodies (or other biological p
When the sample of interest is analysed by ICP-MS in a specialised flow cytometer, each antibody can be identified and quantitated by virtue of a distinct ICP "fo
cell, at a rate of ca. 1,000 cells per second. Because elements are easily distinguished in ICP-MS, the problem of compensation in multiplex flow cytometry is effe
Metal speciation[edit]
A growing trend in the world of elemental analysis has revolved around the speciation of certain metals such as chromium and arsenic. One of the primary techn
chromatography (HPLC) or field flow fractionation (FFF). There are many advantages, from a clinical standpoint, in knowing the specific species present within a
needed by the body and causes no ill effects; however, hexavalent chromium (Cr(VI)), is very toxic to the body. Chromium VI can cause mutations that may lead
It is also beneficial for a clinician to utilize speciation analysis from a preventative standpoint. Many patients, who suffer from elevated levels of certain metals, d
a physician can better narrow the search for possible exposure sites, therefore helping the patient to avoid certain areas in the future.[citation needed]
Quantification of proteins and biomolecules[edit]
There is an increasing trend of using ICP-MS as a tool in speciation analysis, which normally involves a front end chromatograph separation and an elemental sele
chromatography and quantitative preparative native continuous polyacrylamide gel electrophoresis (QPNC-PAGE) for identifying and quantifying native metal co
Recently[when?] a new type of protein tagging reagents called metal coded affinity tags (MeCAT) were introduced to label proteins quantitatively with metals, e
of proteins or other biomolecules like peptides. MeCAT comprises a site-specific biomolecule tagging group with at least a strong chelate group which binds met
amount of analyte which is at least 2–3 orders of magnitude more sensitive than other mass spectrometry based quantification methods. By introducing several
zeptomol range are within the realm of possibility. By using different lanthanides MeCAT multiplexing can be used for pharmacokinetics of proteins and peptides
Breakable PAGE SDS-PAGE (DPAGE, dissolvable PAGE), two-dimensional gel electrophoresis or chromatography is used for separation of MeCAT labelled proteins
performed by dissolving the DPAGE gel after electrophoresis and staining of the gel. MeCAT labelled proteins are identified and relatively quantified on peptide l
Elemental analysis[edit]
The ICP-MS allows determination of elements with atomic mass ranges 7 to 250. This encompasses Li to U. Some masses are prohibited such as 40 due to the ab
dimer), and mass 56 (due to ArO), the latter of which greatly hinders Fe analysis unless the instrumentation is fitted with a reaction chamber.
A typical ICP-MS will be able to detect in the region of nanograms per litre to 10 or 100 milligrams per litre or around 8 orders of magnitude of concentration uni
Unlike atomic absorption spectroscopy, which can only measure a single element at a time ICP-MS has the capability to scan for all elements simultaneously. This
spectrum from lithium to uranium in every analysis won the Silver Award at the 2010 Pittcon Editors' Awards. (http://en.wikipedia.org/wiki/Inductively_coupled

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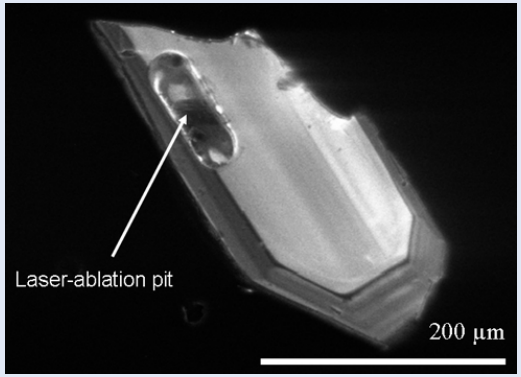
LA-ICP-MS system

U-Pb age determination of accessory minerals

In addition to trace element determination, the LA-ICP-MS system can be used to obtain U-Pb ages in accessory minerals including zircon, titanite, and xenotime.

This example shows a cathodoluminesence image of a zircon grain following U-Pb age determination. The laser has been scanned along a line producing an elongated crater.

It is possible to obtain multiple ages for events in a single crystal where core-rim or other growth domains are observed.



Key instrument capabilities

This dedicated LA-ICP-MS system is capable of low level trace element determinations in most solid materials with minimal sample destruction and spatial resolutions of 5 to 150 microns.

Precise U-Pb age determinations can be obtained in zircons and other accessory mineral phases. Material may be prepared as grain separates or in situ age determinations may be obtained from petrographic sections.

How it works

A laser beam is focused and fired at the sample surface.
As the laser drills into the sample, the material removed is picked up in a flow of gas
This is transported to an ICP-MS where the material is ionised.
Ions are separated according to their mass/charge ratio and the ions counted.
Quantitative analyses can be obtained for: most elements in the mass range 5-250amu (practically 7Li to 238U).

Typical detection limits: in the order of a few parts per billion (ppb) to a few parts per million (ppm).

To fully quantify data the concentration of an internal standard must be known prior to laser analysis. This is typically the concentration of a major element determined by electron probe micro-analysis (EPMA).

http://www.nhm.ac.uk/research-curation/science-facilities/analytical-imaging/micro-analysis/la-icp-ms/examples/index.html

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LA-ICP-MS system

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