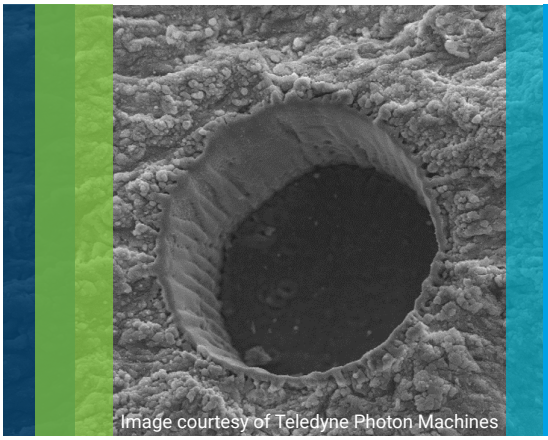

Laser Ablation (LA) ICP-MS Fundamentals

Agilent ICP-MS technology brief

Introduction to Laser Ablation (LA) ICP-MS.

While mainly used to analyze liquid samples, ICP-MS is also used for direct analysis of solids and gases. Laser ablation is the most common approach for solid samples, having been used since the early days of ICP-MS.

LA-ICP-MS uses a laser to produce pulses of high-energy UV light that are focused onto the sample surface using a lens like the one found in a microscope. The laser can be focused into a spot as small as a few µm diameter. The sample is held in an enclosed cell mounted on a movable stage, so the sampling point can be moved beneath the laser beam.

Each laser pulse or "shot" creates a plasma discharge that ablates the sample surface, removing material for analysis. The ablated material is carried in a gas flow (usually He) from the ablation cell through a flexible tube to the ICP-MS. In the argon plasma (the ICP), the ablated sample material is decomposed, atomized, and ionized, before being passed into the mass spectrometer (MS).

Successful LA-ICP-MS analysis requires an ICP-MS with high sensitivity, low background, good matrix tolerance, and effective control of interferences. A wide linear dynamic range is also beneficial as it allows a major element to be used as the internal standard.

Basic requirements for laser ablation ICP-MS

There are many different LA-ICP-MS applications. For each type of analysis, appropriate laser sampling parameters are applied by selecting the laser energy, spot size, repetition rate, and x, y, z position of the stage, together with the ICP-MS acquisition parameters.

Common applications include bulk analysis, where a large sample area is ablated during each acquisition. LA-ICP-MS analysis of a bulk sample produces a steady-state signal that is handled similarly to liquid analysis. Replicate measurements can be collected, and external calibrations used, if suitable solid standards are available.

LA-ICP-MS is also used for microanalysis, where small features/inclusions are ablated individually, and for depth profiling and imaging, where time-resolved analysis (TRA) data is collected as the sample is being ablated. For these types of time-based measurements, acquisition speed is an important factor. However, faster acquisitions use shorter dwell times, giving fewer counts for each analyte. So, for multielement analysis of short-lived TRA signals, a high sensitivity ICP-MS is even more essential.

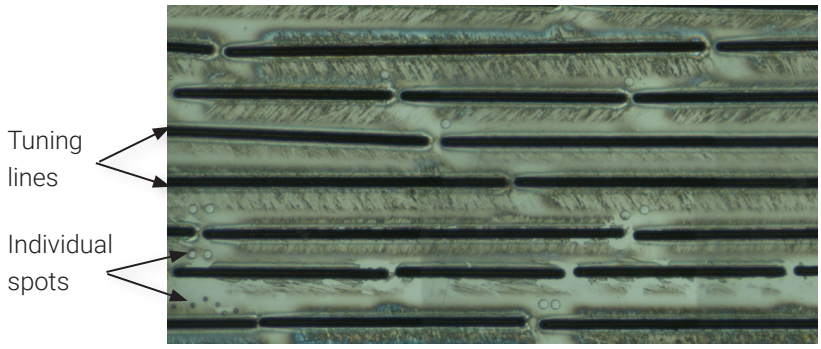

Figure 1. Ablation lines and spots in NIST 612 Glass, used for LA-ICP-MS tuning. Image courtesy of Maxwell Morrisette, CODES Analytical Laboratories, University of Tasmania, Australia.

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Optimizing LA analysis parameters

LA conditions are optimized to produce vapor and fine particles, which must be smaller than ~100 nm to be processed effectively in the plasma. Particle size depends on the sample composition and morphology, and the laser wavelength, pulse width, and energy or fluence (in J/cm^2).

Higher fluence removes more sample mass, giving higher signal. But higher fluence also causes more damage to the sample and creates larger particles, which are not fully decomposed in the plasma, leading to high oxides, poor stability, and elemental fractionation.

Agilent ICP-MS systems give extremely high sensitivity in dry plasma conditions, which allows Agilent LA-ICP-MS users to use fluence around 1 to 2.5 J/cm^2, depending on sample type. This ensures signals are stable for an extended period (as illustrated in Figure 2), while causing minimal damage to small or precious samples.

Plasma robustness is another key factor for LA-ICP-MS performance. The "dry" LA plasma has more energy available for matrix decomposition and analyte ionization, but robustness (indicated by a low oxide ratio) is still critical to performance. The normal CeO/Ce oxide ratio used for solution mode is not accessible when tuning LA-ICP-MS using NIST Glass Standards, as the SRMs contain Gd, which overlaps CeO at *m/z* 156. LA-ICP-MS users therefore monitor plasma robustness using the ThO/Th ratio; a ThO/Th ratio of around 0.001 (0.1%) indicates robust, matrix tolerant LA-ICP-MS conditions.

As with solution mode, if the user disregards plasma robustness, much higher sensitivity can be achieved by using tune conditions such as higher carrier gas flow and shorter plasma sampling depth. But operating with a less robust plasma compromises performance, leading to more drift, higher interferences, and worse fractionation.

Conclusion

Agilent ICP-MS systems have exceptionally high sensitivity in dry plasma conditions, making them ideally suited to LA-ICP-MS applications. The high signal to noise of the Agilent ICP-MS enables accurate analysis at trace levels, even at the low ablation energy (1 to 2.5 J/cm^2) appropriate for small and precious samples. The wide dynamic range detector allows a major element to be used as the ISTD.

LA-ICP-MS line analysis of NIST 610 Trace Elements in Glass SRM.

Agilent 7900 time resolved analysis signals acquired at 30 ms per mass, demonstrating:

- **High sensitivity**
- **Good stability**
- **Low fractionation**
- **Wide dynamic range (majors and trace elements measured in the same acquisition).**

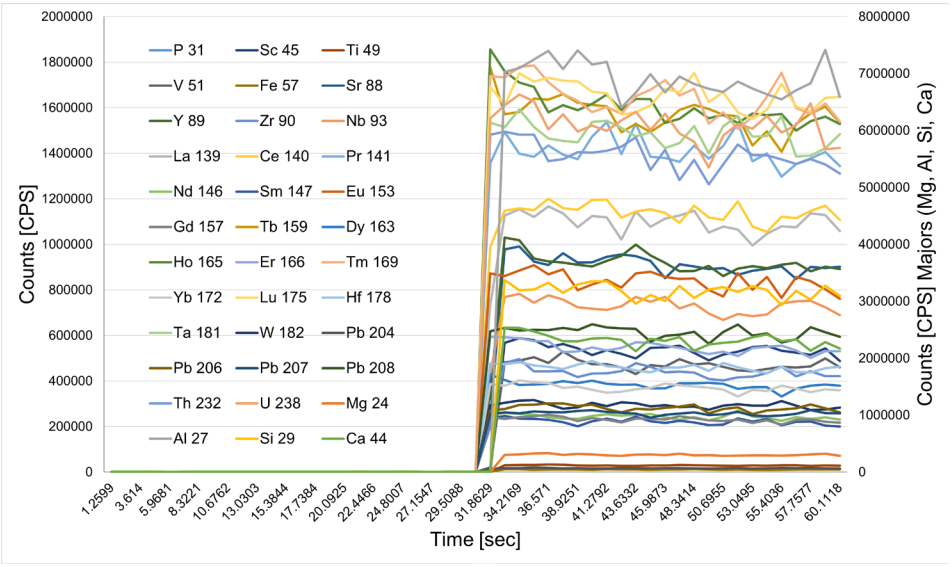

Figure 2. Agilent 7900 signal for 36 masses acquired from a LA line ablation of NIST 610 SRM: 30 seconds of gas blank followed by 30 seconds ablation. 193 nm Excimer laser; 40 µm circular spot, 5 Hz repetition rate at 2 J/cm^2 fluence. Data courtesy of CODES Analytical Laboratories, University of Tasmania, Australia

Learn more:

www.agilent.com/chem/LA-ICP-MS

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