

Applications Note

The TwoVol3 Ablation Cell for NWR Platform

Ultra-fast, -stable and -accurate analysis

A Leap Forward for Two Volume Ablation Cells

TwoVol3 is a brand-new ablation cell for the NWR platform that takes all the advances from the TwoVol2 – the world's most efficient purge, excellent positional reproducibility and fast washout – and adds greater speed and versatility.

TwoVol3 offers two completely different, rapidly interchangeable, ablation cups for the ultimate in application versatility. The Imaging Cup is perfect for ultra-fast imaging and "single shot" applications, whilst the Analytical Cup is ideal for applications requiring signal stability, such as isotope ratio measurements.

Imaging Cup Mode

The ultra-fast Imaging Cup is a close-proximity tube cell. It captures each ablation event and transports it directly into the core of the plasma – via the dual concentric injector (DCI) device – as fast as possible and with minimal mixing. The goal is to transport the ablated material from each laser shot in as short a time as possible, returning to background after each laser pulse. The result is a signal peak width of < 1 ms (FW 0.1 M), allowing signals to be resolved to background at 1000 Hz.



Figure 1. The TwoVol3 ablation cell Imaging mode.

Analytical Cup Mode

The Analytical Cup has a much larger volume than the Imaging Cup to allow a small amount of aerosol mixing from consecutive laser pulses. This delivers a washout time to 1% of 700 ms (tested on NIST612) allowing repetition rates as low as 20 Hz without aliasing. Washout times can be lengthened (using signal smoothers) or shortened (using the DCI device) to tailor the washout time exactly to your application.

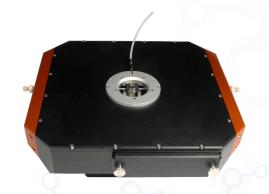


Figure 2. The TwoVol3 ablation cell analytical mode.



TwoVol3 Imaging Cup - How It Works

The new Imaging Cup for the TwoVol3 ablation cell radically alters the geometry inside the cell by reducing the internal volume, adding a cross-flow of He and sealing the top of the volume onto the window. The bottom of the cup is precisely spaced from the sample surface to ensure optimum transport of the ablation plume away from the sample, and ejection directly inside the cup. The sample outlet is via a small-diameter, interchangeable PEEK capillary to ensure the fastest possible transfer time to the ICP.



Figure 3. A closeup view of the Imaging Cup on the TwoVol3 ablation cell.



Figure 4. The TwoVol3 ablation cell in Imaging mode with the DCI.

The chamber interfaces with the ICPMS via Elemental Scientific Lasers' patented dual concentric injector (DCI). This device enables the capillary to continue to the tip of the injector where a concentric flow of Ar gas sheaths the sample into the core of the plasma. A single capillary is used to carry the ablated material from the Imaging Cup directly to the tip of the injector with no interruptions to the smooth flow, and eliminate mixing of the ablated material.

The combination of the Imaging Cup and DCI concepts result in the fastest peaks available: < 1 ms from baseline—peak—baseline. This can be used for single m/z analysis in sequential mass spectrometers (e.g. ICP-QMS; ICP-SF-MS). But in a fast pseudo-simultaneous ICP-MS like time-of-flight (ICP-TOF-MS), where many full mass spectra can be recorded per peak, each single ablation results in a single peak that is translated to a single pixel with zero blurring from other pixels. In this combination, the TwoVol3 enables multi-elemental imaging at up to 1000 pixels per second.

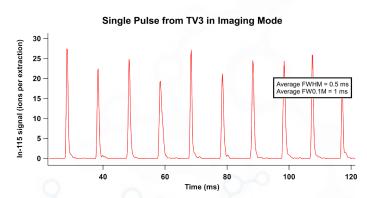


Figure 5. Fast transient peaks captured on a ICP-TOF-MS using the TwoVol3 in Imaging mode.



TwoVol3 Analytical Cup - How It Works

The Analytical Cup for the TwoVol3 ablation cell takes everything we learned about stable and reproducible sample transport from the TwoVol2 and applies it to the concept of switchable geometry. For the first time, it is possible to tune the washout from < 1 ms up to > 2 s by switching cups and adding accessories. In the Analytical Cup, ESL has retained a similar cup volume to TwoVol2 as this allows controlled mixing of the ablation plume between pulses, thereby smoothing the signal for optimum analysis by sequential ICPMS and for the most stable isotope and elemental ratios.



Figure 6. A closeup view of the Analytical Cup on the TwoVol3 ablation cell.



Figure 7. A side view of the TwoVol3 ablation cell showing the sample exit point.

The outlet from the cup feeds directly through a fixed, stainless-steel exit tube, ensuring consistent performance whether your ICPMS is to the left or the right of the laser ablation system. The cup and the objective lens are fixed relative to one another, so complex alignment mechanisms are not required. The exit point from the chamber is static, eliminating potential for leaks that occur through a sliding/rotating seal. The constant position of the cup relative to the inlet ensures consistent gas flow geometries around the ablation site. When coupled with ESL's Typhoon purge, this ensures reproducible and high precision performance, regardless of sampling position.

Switching between the Imaging Cup and the Analytical Cup takes just two minutes and can be done by any operator. Spare cups and windows can be purchased to avoid carry-over from contaminating applications.



Purge Performance of TwoVol3 Ablation Cell

Whichever cup mode is used (imaging or analytical) the same purge process occurs. The class-leading Typhoon purge system, developed by ESL for the TwoVol2 sample chamber, uses its unique flow diffusers to allow He to enter the cell as a smooth, laminar curtain that minimizes mixing and utilizes the differential in gas densities to smoothly push the atmospheric gases down through the exit port. Chambers that rely on vacuum/purge or mixing have been shown to never reach a true background level of O_2 and O_2 , resulting in drift in analytical data. By utilizing a "slow push" approach, Typhoon actually stabilizes background levels in less time than more aggressive methods.

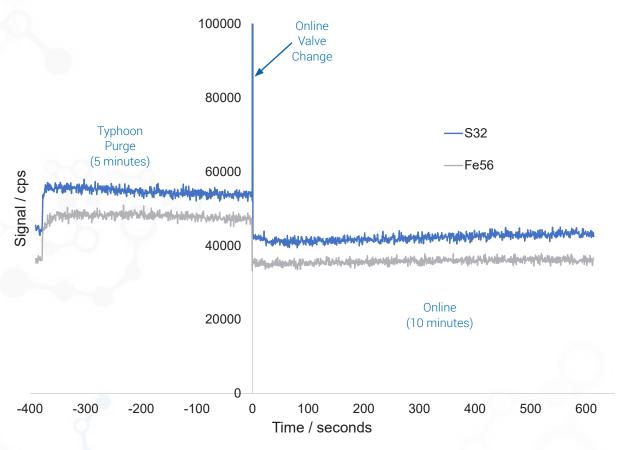


Figure 8. Purge performance of the TwoVol3 (imaging mode) after 5 minutes of purge time.



Spatial Reproducibility of Signal in the TwoVol3 Ablation Cell

The design of the new TwoVol3 permanently maintains the gas flow pathways from the He diffusers to the cup, at every location across the sample surface. Combined with the Typhoon purge's high efficiency at eliminating pockets of air in the cell, the best spatial reproducibility of signals in any ablation cell to date are achieved.

In this experiment nine epoxy mounts, each containing a piece of NIST612 and NIST610, were evenly spaced around the ablation cell. A line scan of 1 mm was placed on the surface of each NIST612 and a transient profile recorded in a single data file. A mean value was calculated for each line scan, and the %RSD of this dataset is reported in Table 1. Most m/z measured gave < 1.5 %RSD.

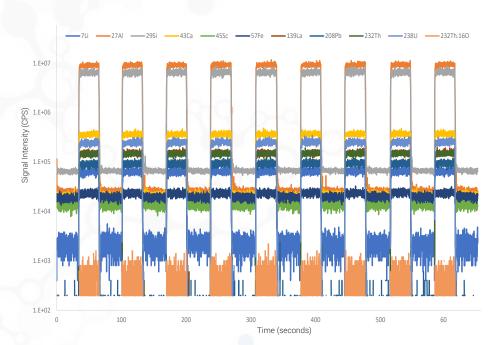


Figure 10. Transient data (single acquisition) from nine line scans on nine separate NIST612 reference standards.

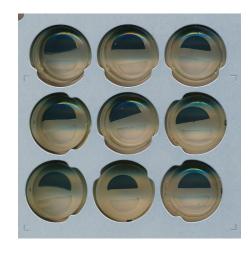


Figure 9. Sample insert for the TwoVol3 ablation cell with nine 1" epoxy mounts (NIST612 and NIST610).

Table 1. Precision data from Figure 10 showing that point-to-point data reproducibility is < 1.5% RSD.

Isotope	%RSD	
⁷ Li	1.35%	
²⁷ Al	1.13%	
²⁹ Si	1.36%	
⁴³ Ca	1.14%	
⁴⁴ Ca	0.83%	
¹³⁹ La	1.06%	
²⁰⁴ Pb	1.39%	
²⁰⁸ Pb	1.36%	
²³² Th	1.41%	
238U	1.49%	



Imaging Using the TwoVol3 Ultra-fast Imaging Cup

The Imaging Cup is designed to couple with simultaneous ICPMS systems, especially time-of-flight (TOF), since they have the analytical speed (up to 36,000 full mass spectra per second) or fast sequential ICPMS in single m/z mode to accurately monitor and integrate each pulse of the laser resolved to background at up to 1000 Hz.

Using the imageBIO266 equipped with TwoVol3 and the icpTOF-2R at LGC (Teddington, UK) sections of mouse brain tissue (courtesy of Northwestern University) were imaged at a resolution of $10 \mu m$. The sample size was $5.5 \text{ mm} \times 4.5 \text{ mm}$ and total imaging time was 41 minutes. Figure 12 shows overlaid distributions of 56 Fe (red) 64 Zn (green) and 63 Cu (blue).

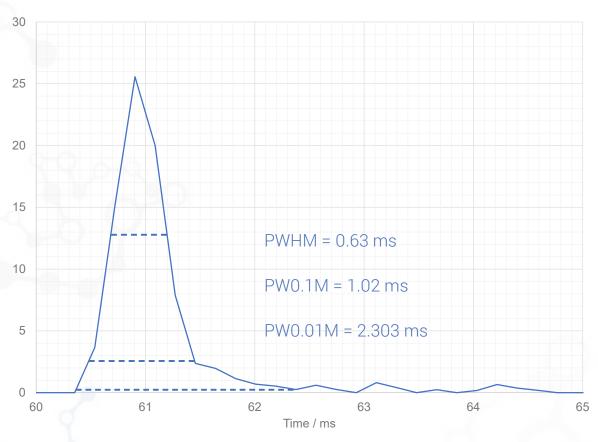


Figure 11. Zoomed in view of a single fast peak (115 ln) created using the TwoVol3 cell in Imaging mode. Full width, half maximum takes just 0.63 ms, and resolution of the peak at 10% is just 1.02 ms.



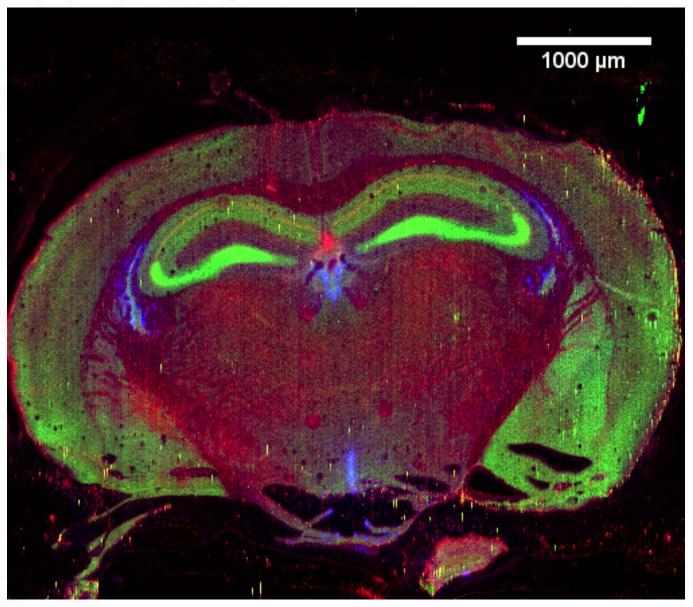


Figure 12. Cross-sectional imaged of a mouse brain, acquired in just 47 minutes by LA-ICP-TOF-MS using the TwoVol3 ablation cell on a imageBIO266.

Benefits of TwoVol3

- Imaging Cup reduces peak widths down to < 1 ms (FW 0.1 M) for high-speed imaging applications.
- Analytical Cup gives washout speeds of 700 ms (to 1%).
- Takes two minutes to switch between imaging and analytical modes.
- Washout time tunable from < 1 ms to > 2 s for application-specific performance.
- Typhoon purge mechanism achieves background levels of atmospheric gas in a few minutes.
- 3 axis linear, closed-loop stage unit gives 10 nm resolution and 100 nm precision for perfect return-to-position performance.