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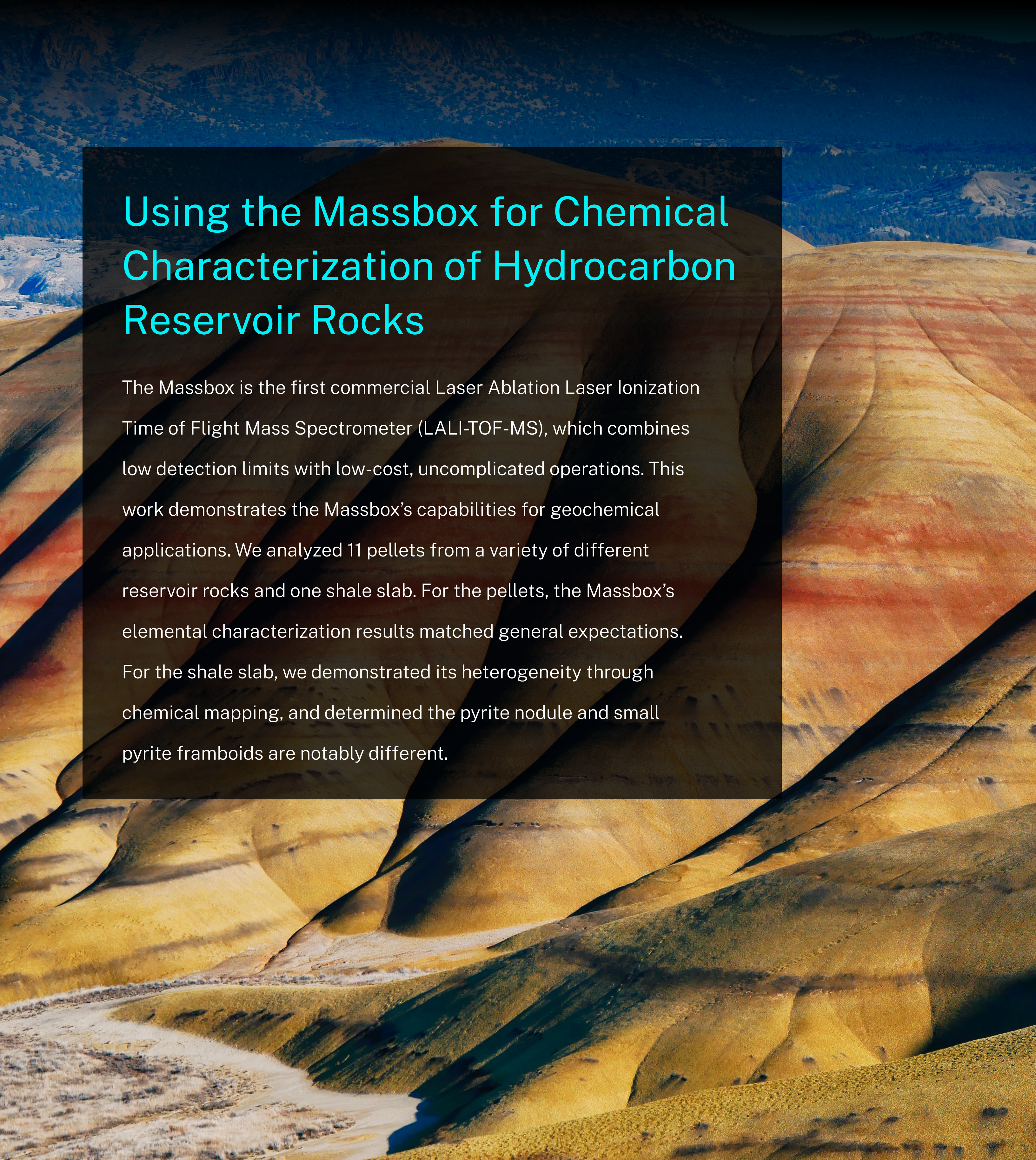
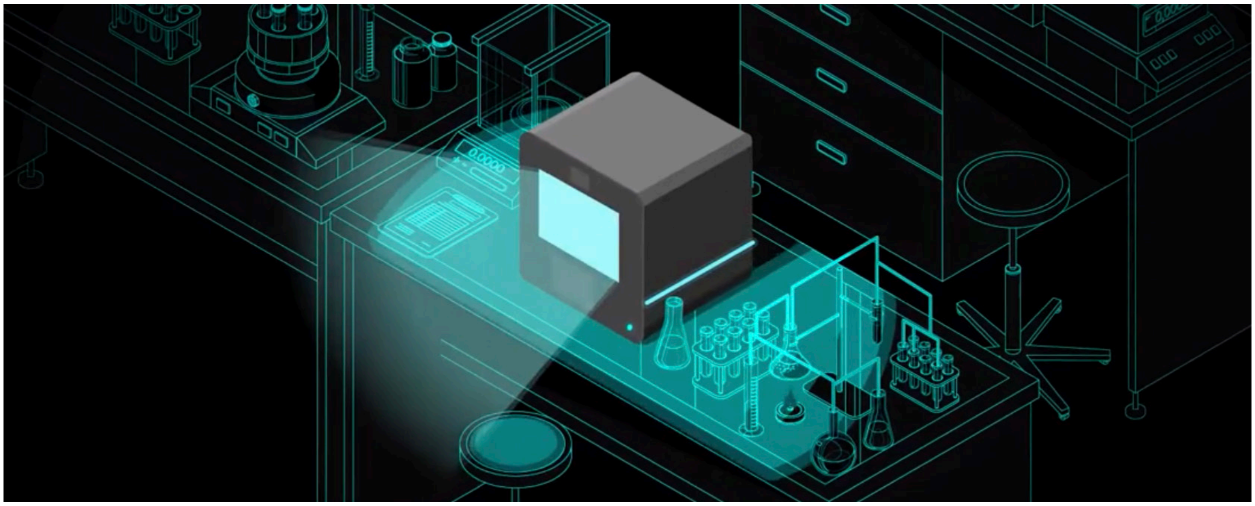


Using the Massbox for Chemical Characterization of Hydrocarbon Reservoir Rocks

The Massbox is the first commercial Laser Ablation Laser Ionization Time of Flight Mass Spectrometer (LALI-TOF-MS), which combines low detection limits with low-cost, uncomplicated operations. This work demonstrates the Massbox's capabilities for geochemical applications. We analyzed 11 pellets from a variety of different reservoir rocks and one shale slab. For the pellets, the Massbox's elemental characterization results matched general expectations. For the shale slab, we demonstrated its heterogeneity through chemical mapping, and determined the pyrite nodule and small pyrite framboids are notably different.

Introduction

For oil and gas extraction, understanding the chemical composition of reservoir rocks is essential. Such elemental and mineralogic data can inform optimized well drilling or completion strategies to maximize production. Targeting the best reservoir rock requires knowledge of not only its chemical properties but at what scale these properties change. One way to gather such information is well logging, a process of running radioactive or sonic tools in the drilled wellbore. While helpful, logging data is not comprehensive. Extracting rock pieces during drilling (i.e., cuttings) or after drilling (i.e., cores) allows direct analysis, which yields more accurate and precise measurements.



LALI-TOF-MS

LALI incorporates two lasers to first ablate (or desorb, in the case of organics) material from the solid sample's surface and then ionize that material in a second step. By analyzing solid samples directly, LALI eliminates the intricate dissolution/digestion sample preparation procedures that complicate other methods. The initial ablation (or desorption) process creates both a temporal plasma and a neutral particle cloud, and the second laser ionizes the neutrals. Compared to other plasma-ionizing techniques, targeting neutral particles greatly reduces the matrix effects.

After ionization, the particles move through the optics system to the Time of Flight (TOF) mass analyzer, which measures the time required for ions of different masses to impact a detector. The resulting measurement creates a full mass spectrum, which facilitates multielement quantitation. Additionally, from ablation to mass analysis, the sample and its representative ions are under vacuum, which improves ion transport efficiency compared to other techniques.

METHOD AND MATERIALS

Premier Oilfield Group (POFG) provided 11 samples of pulverized and pelletized rock and one thick shale slab sample. Of the pellet samples, one is clean limestone, six are shale, and four are a combination of dolomite, calcite, and anhydrite. Our collaborators supplied known composition values for three different pellet sample types. Using the known values as standards, we determined the unknown compositions of the other eight samples. Additionally, POFG was interested in measuring the pellets' degrees of heterogeneity. Each pellet's analysis involved three areas (12,000 individual mass spectra each) which were rastered twice and averaged. To exclude any potential surface contaminants, we used only the second passes of each area for the quantitation. Exum's software picked peaks from the average, baseline-subtracted, calibrated mass spectra, and verified assignments using known isotopic ratios. Peak areas were then normalized to a major matrix element for quantitation. Standard deviations quantified heterogeneity across the three raster areas.

The twelfth sample, a piece of shale with a pyrite nodule and small pyrite framboids, was much more heterogenous than the pellet samples. Thus, we chose a larger raster map area to image its composition changes. For the first pass, we used a relatively large laser spot size for coarse resolution and quick run time. After identifying the most heterogenous areas, we investigated those with smaller raster areas at higher resolution (i.e., smaller laser spot size).

Results

The resulting elemental compositions of the pellet samples generally matched POFG's expectations. For the shale and clean limestone pellets, POFG had detailed background data from Inductively Coupled Plasma Mass Spectrometer (ICP-MS). On the other hand, the three dolomite/calcite/anhydrite samples had measurements from a less sophisticated analytical technique, X-Ray Diffraction (XRD). On these samples, the Massbox identified several trace elements that were not measured by XRD. These results confirmed the Massbox successfully characterized the rock samples' expected elements and recognized trace elements unidentified by other techniques. Figure 1 shows example compositions across the various samples. The error bars are standard deviations from each sample's triplicate measurements, which provide a quantified means to evaluate each pellet's heterogeneity. Data points with larger error bars are less homogeneously mixed pellets.

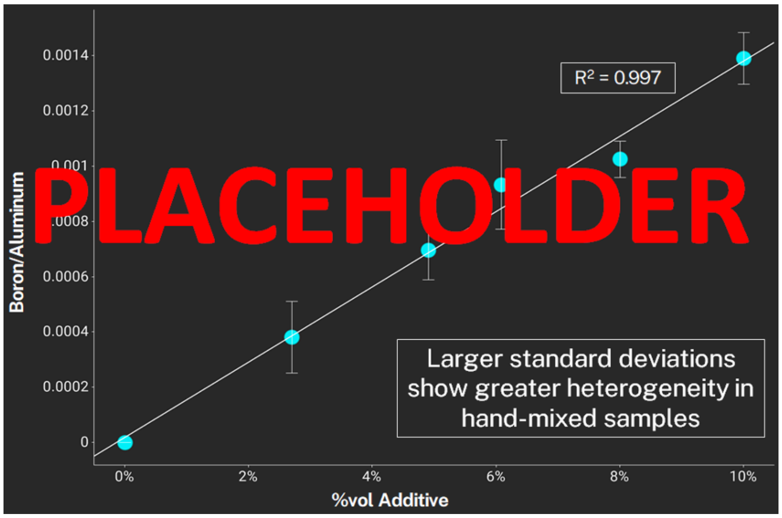


Figure 1: Elemental compositions of several samples. Error bars show the standard deviations from triplicate measurements for each sample.

Results (cont'd)

Figure 2 shows mapping results from the shale slab sample. In the image, each pixel represents one laser-ablated data point. The color scale indicates the measured signal intensity of a particular isotope, with warmer colors designating higher intensities and cooler colors showing lower ones. The image clearly identifies the sample's pyrite nodule and smaller pyrite framboids. As indicated by the different colors of each, there are significant chemical differences among the pyrite-rich areas.

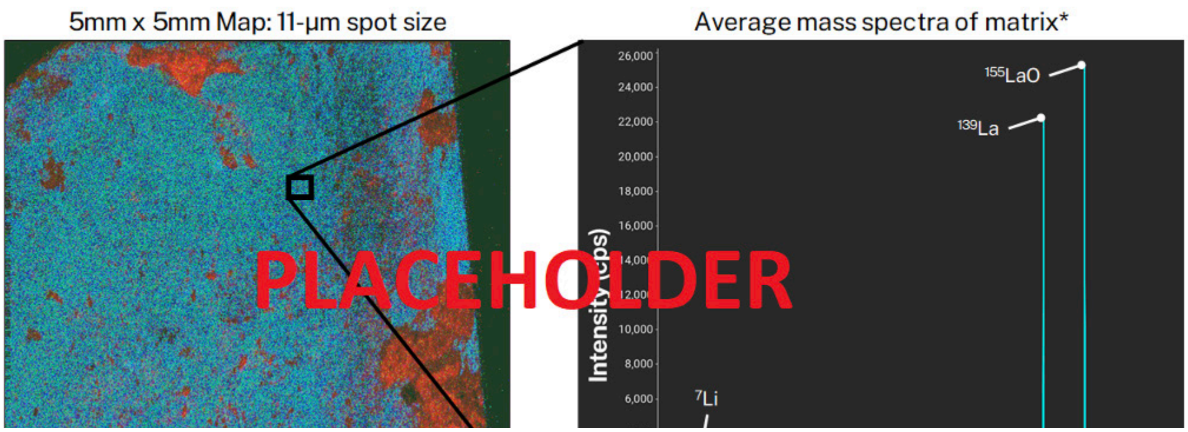


Figure 2: Mapping results from the shale slab sample. The color scale represents a particular isotope with warmer colors designating higher concentrations and cooler colors showing lower.

Conclusion

With the intuitive operations of its all-in-one system, the Massbox is the ideal instrument for geochemical characterization of hydrocarbon reservoir rock samples. Its capabilities provide accurate quantification of important elemental compositions, degrees of homogeneity in pelletized samples, and mapped heterogeneity across a complex sample. With simple sample preparation procedures and a few minutes for analysis and quantitation, the Massbox offers rapid turnaround for geochemical characterization.