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Towards Practical LA-ICP-MS Protocols for Uranium Analysis in UO_2 Fuel Pellets and Beyond

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Conventional uranium(U) analysis of UO_2 fuel pellets is typically performed by wet-chemical dissolution followed by solution-based mass spectrometry, which is labor-intensive, generates radioactive and chemical waste, and requires extensive sample preparation. In this contribution, we investigate laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) with a 193 nm excimer laser coupled to a single-quadrupole ICP-MS as a more rapid, solid-state alternative for U analysis in UO_2 pellets, with the longer-term goal of applying similar approaches to other solid U-bearing materials. Sintered fuel pellets, including compositions with and without Gd_2O_3 additions, were prepared as polished epoxy-mounted cross sections for reproducible ablation.

At this stage, the focus is on identifying practically applicable operating conditions as a basis for establishing a future routine protocol. Laser fluence, spot diameter, and nebulizer gas flow rate, which control the particle injection rate, were varied over a broad range to evaluate their effects on signal intensity, short-term stability, washout behavior, and apparent fractionation during ablation and aerosol transport. Under selected conditions, both spot and line/raster ablation modes provide stable ^{235}U and ^{238}U signals with rapid stabilization that are suitable for time-efficient pellet analysis. Different calibration strategies were examined, including external glass and oxide standards with normalization to ^{238}U and UO_2-based solid reference materials where available, with emphasis on day-to-day robustness in the absence of fully matrix-matched UO_2 standards.

Preliminary comparisons between LA-ICP-MS results and bulk U determinations from wet-chemical analysis indicate that, for undoped UO_2 pellets and optimized ablation conditions, agreement within combined uncertainties is achievable despite the limitations of single-quadrupole mass spectrometry. For Gd_2O_3-bearing fuels, however, larger deviations are observed, reflecting stronger matrix effects, changes in ablation behavior, and transport-induced fractionation that cannot yet be fully corrected using the current reference materials. These discrepancies define the limits of accuracy and point to the need for better-characterized, compositionally matched UO_2-based reference materials and further refinement of instrumental and data-processing protocols.

Overall, this exploratory work summarizes the current operating window, calibration approaches, and limitations of LA-ICP-MS for U analysis in UO_2 pellets and provides a basis for further protocol development and extension to other solid U-bearing materials.