

Analytica-2015 : Precise analysis of elements in silica powders by LA-ICP-MS - Istvan Halasz and Runbo Li - PQ Corporation

Istvan Halasz and Runbo Li

PQ Corporation, USA

Silica is important component of many heterogeneous catalysts. Catalysis affects ~90% of all chemical products. For elemental analysis by AA or ICP these silica-based catalysts are dissolved by HF and other acids. HF is exceptionally dangerous because of its ability to diffuse through the skin where the fluoride ion binds calcium with subsequent disruption of electrical activity. Therefore, it is desirable to minimize its use to reduce risk and eliminate a lengthy and tedious dissolution process. We speculated that coupling laser ablation (LA) to an ICP-MS could fulfill this desire. However, LA cannot be used for powder samples and LA-ICP-MS is known to be much less accurate than liquid phase measurements, owing to inhomogeneous ablated particle size and inadequate analysis parameters. Here, we report the development of a LA-ICP-MS method for accurately analyzing powdered silica supported catalysts. We melt the powders with a mixture of $\text{Li}_2\text{B}_4\text{O}_7$ - LiBO_2 into a homogeneous solid bead, vaporize the surface with a laser, and then apply a small cyclone before the ICP. Moreover, we optimized the analysis conditions by using statistical experimental design of 11 parameters. Using three commercial zeolite catalysts having Si/Al ratios 2.6, 40, and 140, we show that different parameters significantly affect the accuracy of measuring their Al contents. The relative standard deviation, RSD, remains <5% over the entire concentration range tested, sometimes even <0.5%, which is better than that obtained by the HF dissolution technique.

Laser removal inductively coupled plasma-mass spectrometry (LA-ICP-MS), which is a quickly creating expository method for the examinations of follow components and isotopes, assumes a significant job in propelling the investigation of Earth science, particularly as for miniaturized scale geochemistry. This article surveys the use of LA-ICP-MS in the natural examination of strong geographical examples. In spite of the fact that LA-ICP-MS has been broadly utilized in the spatial goals investigation of component creations and fast mass examination of entire stone and soil tests, the

investigation precision is limited by various elements, including the instrumental conditions, the natural fractionation and network impacts, the absence of adequate lattice coordinated reference materials, and the methodologies for quantitative adjustment and affectability float revision. As indicated by the kind of tests and the analyte components, the examination exactness can be improved through the streamlining of instrument conditions and the reception of reasonable rectification techniques and reference materials.

The total substance investigation of regular silicates has recently been cultivated distinctly by consolidating laser removal ICP-MS with electron microprobe (EMP) examination to give corresponding data on significant component constituents. Here, we present a strategy for laser removal ICP-MS examination of significant components in silicate glasses and minerals that, when applied utilizing a 193 nm laser framework coupled to an ICP-MS, gives exactness and precision practically identical to that of EMP procedures. Imitate examinations of USGS glass reference materials BCR-2G and BHVO-2G have inner exactness of 1–4%, and are precise to better than 5%. Further, the LA-ICP-MS technique was applied to volume-found the middle value of investigation of a heterogeneous silicate to yield creations that are more exact than EMP and increasingly precise for minor components, especially MnO. The exactness and accuracy of basic investigations performed with LA-ICP-MS are restricted by numerous elements, including the laser removal conditions, the ICP-MS scientific conditions, instrumental affectability float, grid impacts between the example and measures, fractionation impacts, the precision of the suggested qualities for reference materials utilized for adjustment, and the inside standard utilized for standardization.

Biography:

Istvan Halasz obtained his PhD in Hungary from the Hungarian Hydrocarbon Institute, where he developed and scaled-up efficient processes for pharmaceutical, fine chemical and petrochemical industries. Later, at the Hungarian Academy of Sciences and at USA Universities, he studied zeolite catalysis, oxide superconductor synthesis, and catalytic fume abatement for automobile exhausts. In the past 16 years, he has investigated the properties of silicates at PQ R&D. He chaired the Philadelphia Catalysis Club; is current President of North-East Corridor Zeolite Association (NECZA); edited a book on catalysis, and authored 110+ book chapters and papers, 7 patents, and 80+ conference presentations. Runbo Li obtained her PhD in Analytical Chemistry from Drexel University in USA. In her thesis, she studied different methods for preparing samples for analysis by MALDI TOFMS and applied these methods to quantify proteins. At PQ R&D, she has focused on analytical method development and characterization research related to silicates, glass beads, amorphous silica gel and zeolites. She has published 18 papers.

Email: istvan.halasz@pqcorp.com