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Liquid standard addition calibration for laser ablation inductively coupled plasma optical emission spectrometry

Laura Kronlachner[1], Lisa Lenz[1], Alexander Holzer[1], Aexander Opitz[1], Andreas Limbeck[1]

[1]TU Wien, Institute of Chemical Technologies and Analytics, Wien, Österreich

The analysis of stochiometric ratios in solid-state samples is crucial in various fields when materials with a defined structure are employed for different applications. The stochiometric composition impacts the material's properties, such as reactivity and stability. Especially in applications in electrochemistry, like high-performance ceramics used in cathode materials of solid oxide fuel cells, the stochiometric composition is vital to meet the high demands imposed on the material.

Conventional methods for the determination of the elemental composition of solid samples include the dissolution and subsequent analysis with ICP-MS or ICP-OES. Problems can occur with samples that cannot be dissolved in sufficient quantity. Solid sampling with LA is desirable, but due to occurring matrix effects, reliable quantification is often impaired. Hence matrix-matched standards are necessary, which are not available for most samples. The preparation and characterization of in-house matrix-matched solid standards have the drawback of being challenging and time-consuming.

Thompson et al. [1] developed a method for element concentration analysis with LA-ICP-OES using an aqueous solution as an external standard, applying a dual gas flow inlet system. Further developments allowed mixed-sample introduction [2] and were applied for on-line additions calibrations [3]. In this work, a multiple standard addition approach using liquid standards to create a matrix-matched LA-ICP-OES calibration was employed and applied for inert metal oxides. While absolute quantification of elements requires knowledge about ablation rate and transport efficiency, this is negligible in the case of stoichiometry determination if no fractionation occurs. For the analysis, the solid sample was ablated with a 213 nm laser and the resulting sample aerosol was mixed with aerosol carrying the aqueous calibration standards from a nebulizer before entering the ICP-OES. This simultaneous introduction results in the aqueous standards being analyzed within the sample matrix. The concentration of the standard solution was stepwise increased, creating a matrix-matched standard addition calibration which allows the signal quantification and thus stochiometric determination of electrochemical cathode materials for solid oxide fuel cells.

Laser ablation, Standard addition calibration, Matrix-matched standards, Sample stochiometry

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