Strategy for Semi-Quantitative Direct Gas Analysis Using GC-ICP/MS ¹ National Energy Technology Laboratory, Morgantown, WV 26507 ² Oak Ridge Institute for Science and Education, Oak Ridge, TN 37831

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Abstract

Previously published techniques using GC-ICP/MS describe calibration and analysis methods that are valid for liquid phase analyte injection only due to the limited number of gas phase standards and sampling methods. The few gas phase calibration standards potentially available suffer from poor mass distribution (most are low atomic weight elements), high toxicity (AsH₄, PH₃), and instability or reactivity. Furthermore, generation of a non-reactive gas phase calibration standard containing sub-ppm trace material is technically arduous and multi-element standards are prohibitively expensive. The technical difficulties of gas phase analysis have been addressed for practical gas-phase inorganic analytes by digestion of gas samples in a liquid matrix which remains costly in both resources and time.

This work discusses a semi-quantitative method which can aid in evaluation of gas phase analyte concentrations using direct gas phase injection of sample into the GC-ICP-MS. Semiquantitative procedures are offered by most ICP-MS manufacturers and have been applied to the analysis of both liquid and solid samples in order to determine the elemental compositions of a sample; however, the semi-quantitative procedures for direct gas phase analysis have not been developed. Empirically derived correlations were generated using liquid phase data in order to relate the instrument detector response to analyte mass and plasma conditions for a constant set of instrument operating parameters. Using the methods described in this research, the elemental concentrations in a standard containing 45 analytes and treated as an unknown sample were quantified accurately to +/-50% for most elements using ¹³³Cs as a single internal reference. The method is used to predict liquid phase mercury within 12% of the actual concentration and gas phase mercury within 35% of the actual concentration. The results verify that the calibration method facilitates accurate semi-quantitative, gas phase analysis of metal species with sufficient sensitivity to quantify metal concentrations lower than 1 ppb for many metallic analytes. Such analyses are critical to determine the presence of trace metals in coal derived synthesis gas supplied to a solid oxide fuel cell in advanced electricity generating systems.