Strategy for Semi-Quantitative Direct Gas Analysis Using GC-ICP/MS

PROJECT GOAL: To develop a method that can be used to quantify gas phase contaminants in real time

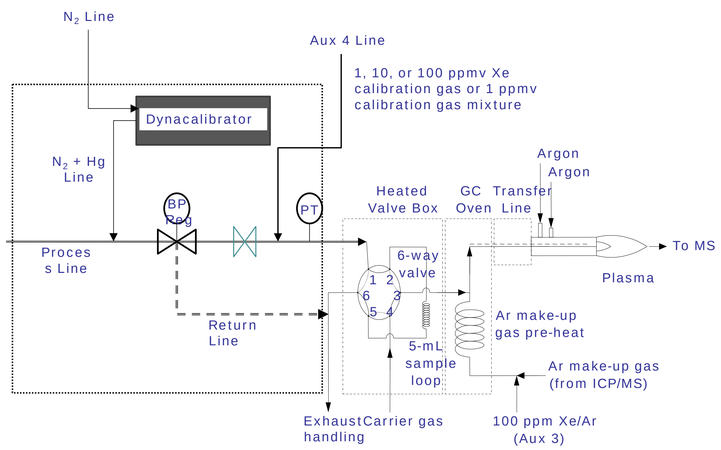
ICP-MS and Industry Standard

- ICP-MS is used to determine trace metal concentration in liquid and solid samples
- Gasification industry uses EPA 29 method for collecting samples and EPA 6020A method for analysis – this can be costly in both time and resources

GC-ICP/MS System installed at NETL

GC-ICP/MS System:

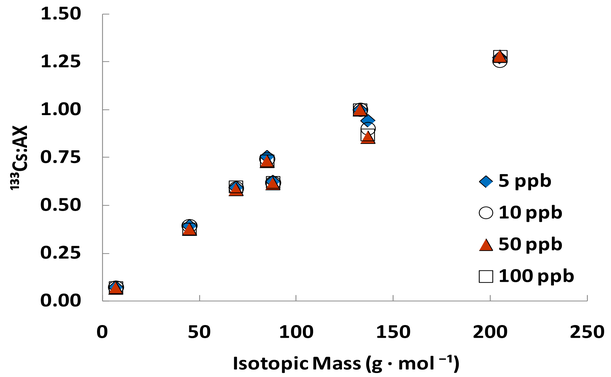
- Accurate to part per billion and part per trillion levels
- Liquid phase semi-quantitative analytical methods are provided by the manufacturer – allows concentration determination within 20 – 30 % of the actual
- No direct gas phase analytical procedure exists – research task EXTENDS ANALYTICAL METHOD TO GAS PHASE SYSTEMS



GC-ICP/MS gas phase injection system

Experimental System

- System was configured to allow direct injection of gas phase samples
- No liquid sampling needed
- Real time sample collection and analysis

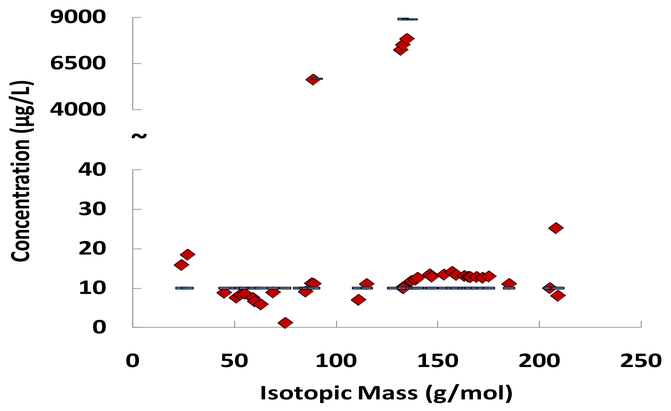


Isotopic Transmission Factors

- Developed using liquid phase samples
- Relates known detector responses for a calibrated element to unknown elements with masses spanning entire periodic table
- Factor used to determine the concentration of different elements in solution using the following equation:

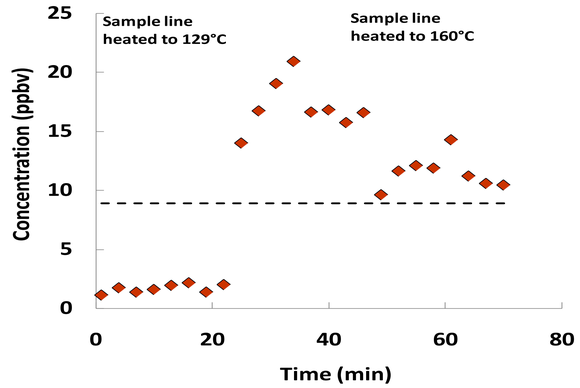
$$e_X = \frac{C_I}{C_X}\frac{N_X}{N_I}\frac{K_X}{K_I}e_I F_X$$

where e_X is the relative ionization efficiencies, N is the response, K is the isotopic transmission factors, C is the concentration of the isotopes ^{Z}I (^{133}Cs) and ^{Y}X, and F_X is a correction factor used depending on the element (i.e. Arsenic, Selenium, or Mercury).



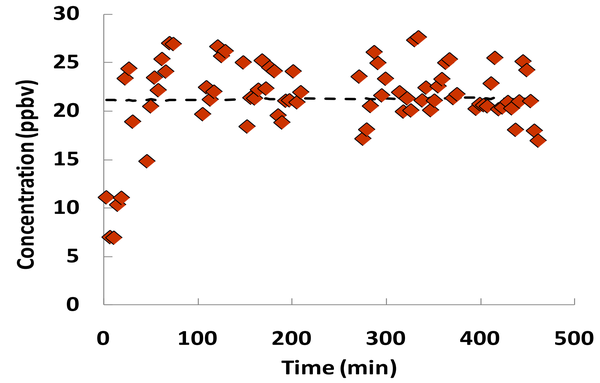
Method Check with Liquid Samples

- Method checked with liquid solutions
- Most elements are within 35% of the actual concentration with the dashed line depicting the actual concentration of each isotope
- Concentrations span 3 orders of magnitude



Temperature Dependence

- Line temperature impacted the transfer of mercury sample to the GC-ICP/MS system
- Lower line temperature allows for adsorption of mercury onto sample transfer line
- Increase in temperature permitted desorption of mercury from the sample line



Gas Phase Sampling of Mercury

- 80 different injections of mercury sample made during the course of 470 minutes (approx. 7.4 hours) – usual sample collection time using EPA method is approximately 9 hours for one sample
- **Predicted mercury concentration was within 5 – 30 % of the actual concentration (depicted with the dashed line)**
- Low predicted concentration at beginning of sampling due to the temperature of sample and transfer line

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