Measurement of Trace Metals in Ambient Air:
Validation of a Rapid Technique with Gold Standard ICP-AES

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Introduction

Trace metals are found at low concentration in the ambient environment and are associated with adverse health effects.

The gold standard metal measurement is Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES); however, sample preparation is laborious and expensive.

The Xact 625 metals monitor uses **non-destructive X-ray fluorescence** technology. A built-in sample collection system enables **near-real-time** trace metal measurements in ambient air. Modifying the Xact monitor to also characterise metal concentrations on externally collected filters would greatly expedite the analysis process and reduce costs.

Research Objectives

(1) Extend the measurement capabilities of the Xact monitor to include filter based metal measurements.

(2) Examine Xact monitor's precision and accuracy of external filter analysis.

Methodology

Metal concentrations were measured using both the Xact Monitor and ICP-AES for three general sets of filter samples: (1) **ambient air**, (2) **a standard reference material (SRM)** and (3) **blanks**. SRM is standardised dust with certificate composition available from NIST. Coal Fly Ash SRM was used in this study.

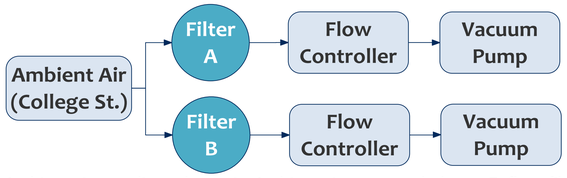

Figure 1: Ambient air sampling schematic. Ambient air was sampled onto Zefluor filters with a flow rate of 10L/min for 24 hours. Two filters were run in parallel on 8 separate days.

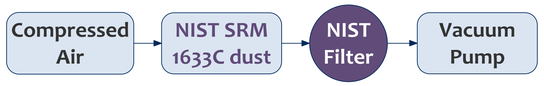

Figure 2: NIST sampling schematic. NIST SRM was loaded onto five Zefluor filters.

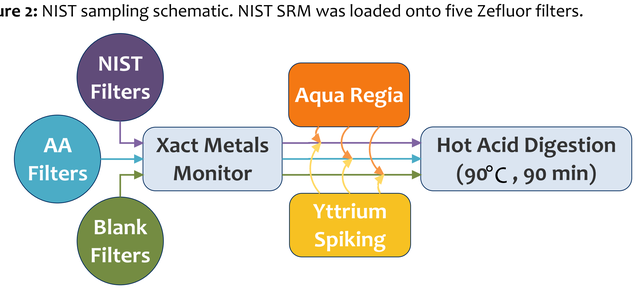

Figure 3: Filter samples analysis. Samples were first run on the Xact and then digested for ICP-AES.

For ICP-AES analysis, filters were extracted through hot acid digestion with an aqua regia solution. The aqua regia solutions were prepared with 30% trace grade HCl and 60% trace grade HNO_3 in a 3:1 ratio. Samples were then heated in a water bath at 90°C for 90 minutes. Residual particles were removed with a Whatman® syringe filter, 1µm pore size. All samples were spiked with 20 ppb yttrium to account for any volume loss during in the digestion process.

Aqua Regia	3.84 ml	15% HNO_3, 22.5% HCl
Yttrium Spiking	0.16 ml	500 ppb Y in 6.5% HNO_3
Total	**4.00 ml**	**14.66% HNO_3, 21.6% HCl, 20 ppb Y**

Table 1: ICP-AES sample volume and composition.

Results

ICP-AES VS. NIST Certificate

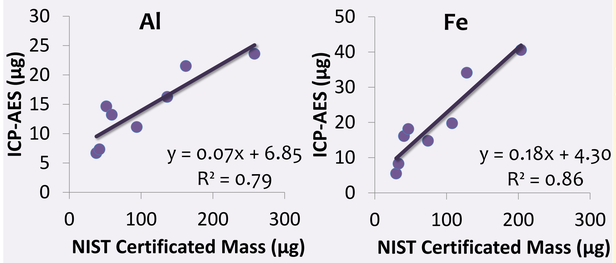

Figure 4: Correlation between ICP-AES results of NIST filters and certificated compositions.

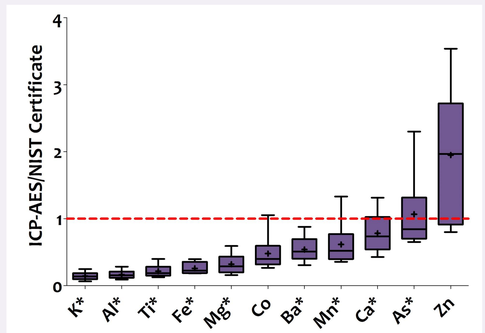

Figure 5: Ratio between ICP-AES results of NIST samples and certificated compositions for selected elements.
*: *R^2 value of correlation for the corresponding element is above 0.6.*

Xact VS. NIST Certificate

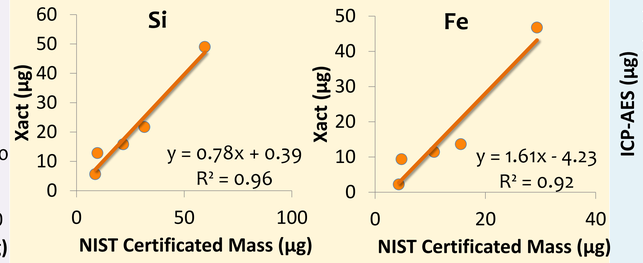

Figure 6: Correlation between the metals concentration of NIST filters given by the Xact metals monitor and NIST certificated compositions.

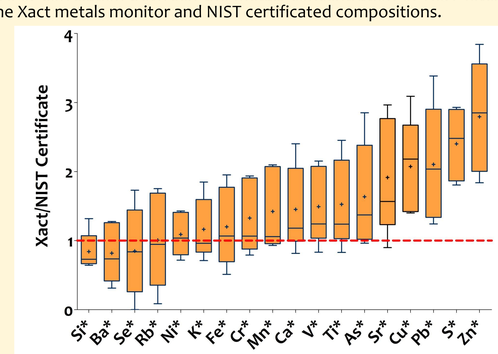

Figure 7: Ratio between Xact monitor's results of NIST samples and certificated compositions for selected elements.
*: *R^2 value of correlation for the corresponding element is above 0.6.*

Xact VS. ICP-AES: Ambient Air Measurements

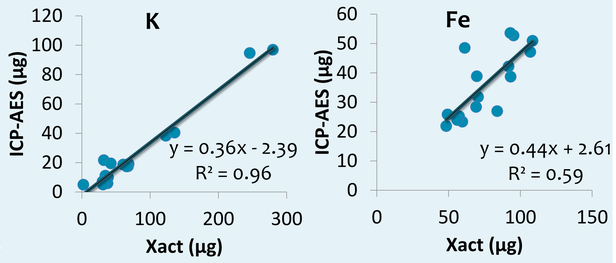

Figure 8: Correlation between the metals concentration of ambient air filter samples given by the Xact metals monitor and ICP-AES.

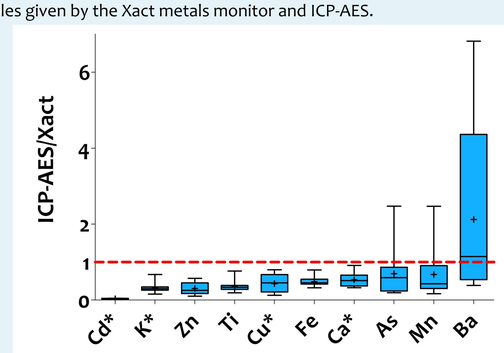

Figure 9: Ratio between ICP-AES and Xact monitor's results of NIST samples for selected elements.
*: *R^2 value of correlation for the corresponding element is above 0.6.*

Summary

Comparison between Xact Metals Monitor and ICP-AES

- Linear regression models showed that the results from the Xact metals monitor has a better correlation with the NIST certificated composition than ICP-AES
- Significant differences have been identified between the results from the Xact monitor and ICP-AES. The Xact metals monitor generally overestimates sample metals concentration whereas ICP-AES underestimates. Follow-up analysis is needed to further investigate the reasons
- Metals analysis of filter samples using the Xact metals monitor takes significantly less time (~20 minutes) than using ICP-AES (90 minutes hot water bath for sample acid digestion, ~5 minutes per sample when analyzing)
- Near-real-time analysis of metals with the Xact monitor allows for potentially improved identification of pollution sources; however, further validation studies are needed

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