Exploring possibilities and drawbacks in the analysis of Pb loaded reference samples by using TXRF spectrometers

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INTRODUCTION

Air pollution sources:
- Dust & Construction
- Waste burning
- Transport
- Diesel Generator
- Industries
- Domestic Cooking

ENVIRONMENTAL MONITORING

- Particulate matter (PM) is a prominent air pollutant responsible for negative health effects
- The EU commission established thresholds in the concentration of potentially toxic elements in air, such as Pb, As, Ni, Cd, and Hg [1]

ANALYTICAL METHODS FOR AIR FILTERS

- Conventional: ICP-MS, AAS
- Novel: SMART STORE\(^{®}\) + TXRF analysis [2]

SAMPLE PREPARATION

- Set of 6 reference samples: 4 Pb-loaded samples, 1 blank and 1 multi-element sample
- Mass deposition ranging from 0.028 to 10.169 \(\mu\)g/cm\(^2\)
- Samples handling with SMART STORE\(^{®}\)

INSTRUMENTATION

- SMART STORE is suitable for air PM filters preparation and handling. Sample can be measured with commercial TXRF spectrometers.
- Linear model is improved
- The mass-absorption contribution was considered using the following equation:

\[
N_x = c_xS_x \frac{1 - \exp[-(\mu/\rho)_{M}d]}{\mu/\rho_{M}} N_0 = c_xS_xN_0k_x
\]

N\(_x\): integrated intensity
\(c_x\): mass fraction
\(S_x\): element sensitivity
\(\mu/\rho_{M}\): mass attenuation coefficient
N\(_0\): intensity of the exciting monochromatic beam

RESULTS AND DISCUSSION

QUALITATIVE ANALYSIS

- Pb detected with all the TXRF spectrometers
- Intensity differences: instrumental configuration and live time measurement

QUANTITATIVE ANALYSIS

1. EMPIRICAL APPROACH

- Empirical calibration approach: net integrated intensities of samples vs. their known mass loading [3].
- Statistical tests: Mandel test and Lack of fit
- Regression model: the quadratic fits better the whole set of samples
- Excluding the sample with higher mass deposition, the linear model is improved

2. THEORETICAL APPROACH

- The mass-absorption contribution was considered using the following equation:

\[
\text{Absorption phenomena}
\]

CONCLUSIONS

- SMART STORE\(^{®}\) is suitable for air PM filters preparation and handling. Sample can be measured with commercial TXRF spectrometers.
- Quantitative analysis can be implemented by external calibration.
- LOD, LOQ and linear range are determined.
- LOD equal to 0.0065 \(\mu\)g/cm\(^2\), well below the EU limits for air quality monitoring.

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REFERENCES