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

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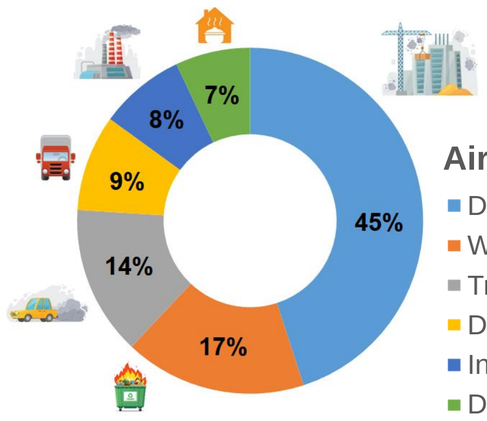
INTRODUCTION



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]

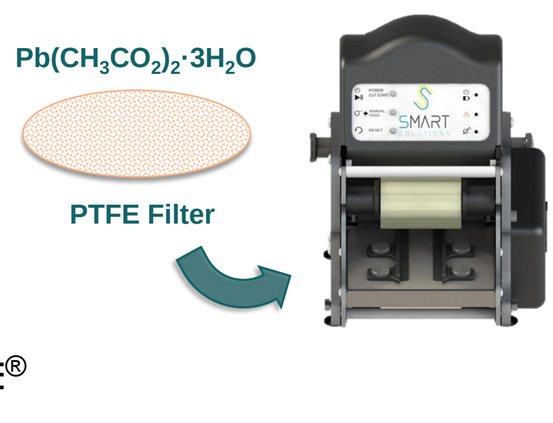
ANALYTICAL METHODS FOR AIR FILTERS

- Conventional: ICP-MS, AAS
- Novel: SMART STORE® + TXRF analysis [2]

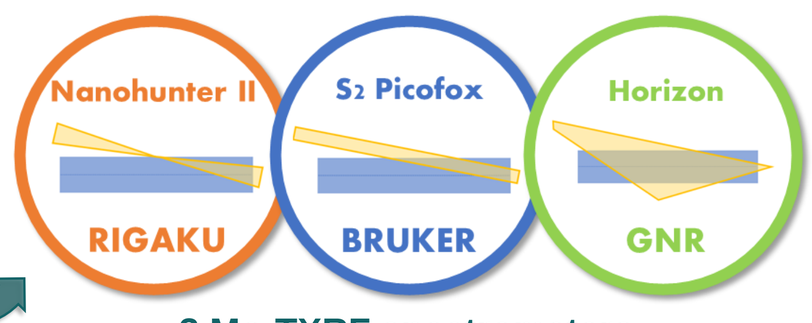
EXPERIMENTAL

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 μg/cm^2
- Samples handling with SMART STORE®



INSTRUMENTATION

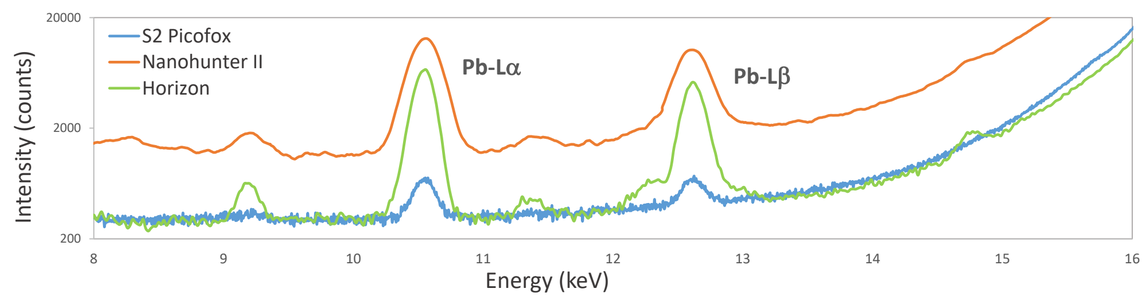


3 Mo-TXRF spectrometers:

RESULTS AND DISCUSSION

QUALITATIVE ANALYSIS

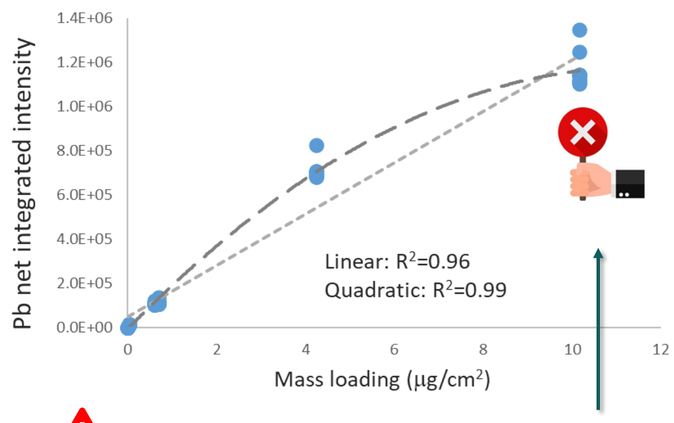
- Pb detected with all the TXRF spectrometers
- Intensity differences: instrumental configuration and live time measurement



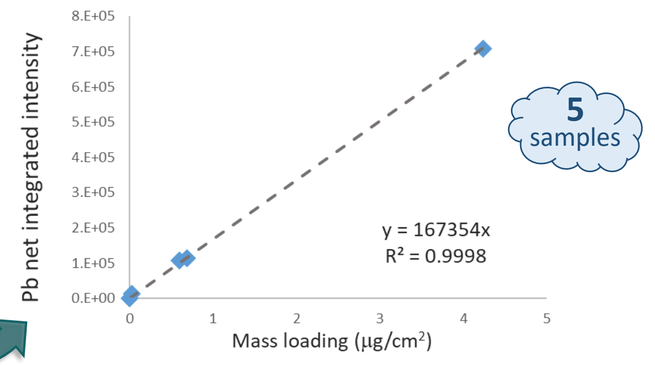
QUANTITATIVE ANALYSIS

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



Absorption phenomena

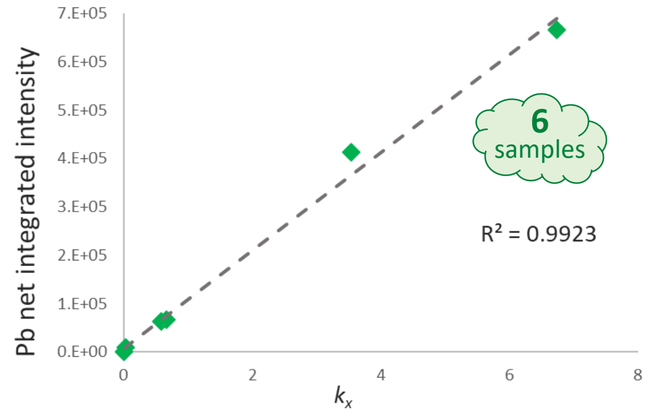


② THEORETICAL APPROACH

- The mass-absorption contribution was considered using the following equation:
- Linearity was observed considering all the six samples

$$N_x = c_x S_x \frac{1 - exp[-(\mu/\rho)_M \rho d]}{(\mu/\rho)_M} N_0 = c_x S_x N_0 \boldsymbol{k_x}$$

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



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 μg/cm^2, well below the EU limits for air quality monitoring.

ACKNOWLEDGMENT

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REFERENCES

[1] Directive 2008/50/EC, Official Journal of the European Union, L 152/1
[2] L. Borgese et al., Spectrochimica Acta Part B 167, 105840, 2020
[3] P. Cirelli et al., Spectrochimica Acta Part B, submitted