

## INTRODUCTION



It is estimated that air pollution is responsible for more than 400 k deaths per year in Europe. Concentration of pollutants in air must not exceed the limits established by EC

Particulate matter (PM) composition can be determined analysing air filters



Conventional methods for filter analysis are destructive, time-consuming and polluting

It is possible to perform a direct analysis of air filters properly prepared, in XRF under grazing incidence condition



Quantitative data can be obtained using external calibration lines, built using reference samples

Reference samples are made nebulizing a solution of known concentration and collecting the aerosol onto a PTFE filter



## SAMPLE PREPARATION

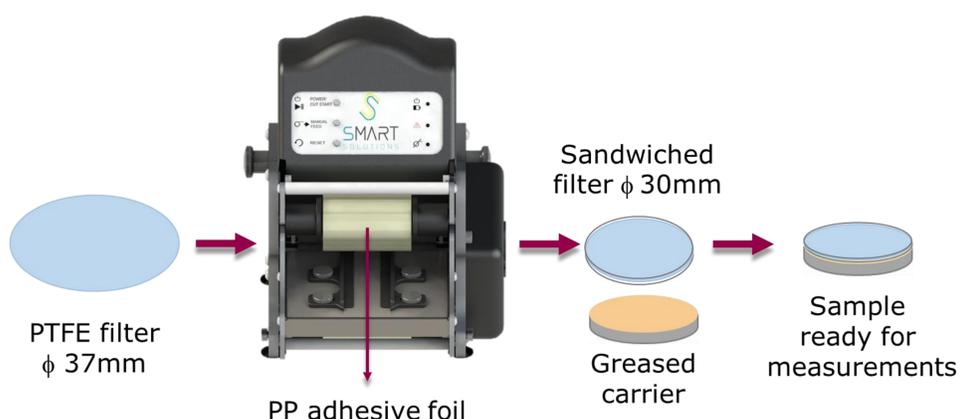
A solution of known elemental concentration is nebulized

The generated aerosol is conveyed into a mixing chamber

A PTFE filter placed into a filter holder collects the aerosol

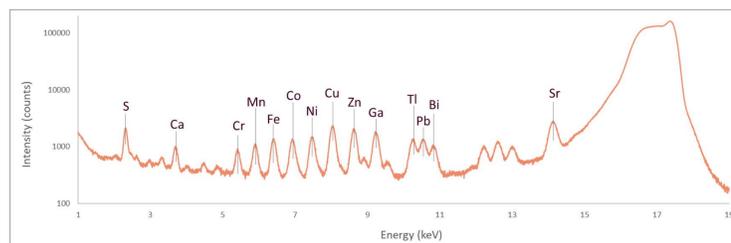
An air pump sucks the aerosol to foster its deposition on the filter

The filter is ready for the **SMART STORE**<sup>®</sup> sample preparation procedure:



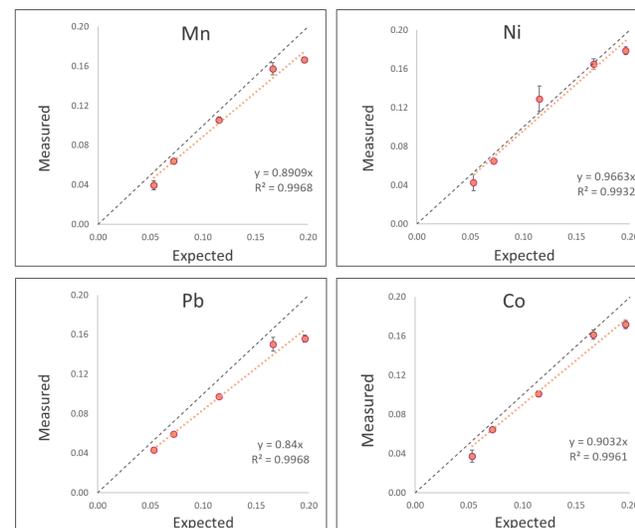
## RESULTS

- The samples under study were created nebulizing a multi-elemental solution (ICP IV, 23 elements). The mass loading of the samples was selected in a range close to the concentration limit imposed by EC for air quality [1].
- Surface concentration of the sample with smaller mass corresponds to an elemental concentration in PM equal to 0,01 µg/m<sup>3</sup>.
- 5 samples with different surface concentration were realized and measured in XRF under grazing incidence condition, with the TXRF spectrometer S2 Picofox (Bruker), equipped with a Mo X-ray tube.



Spectrum of a sample exposed for 20 minutes to a nebulised solution of ICP IV. The main elements are identified. The X-ray source is Mo (17,5 keV).

- Preliminary results show a good accordance between the expected surface concentration and the experimental values obtained by internal standard (Ga) calibration.



Average surface concentration [µg/cm<sup>2</sup>], obtained considering 4 measurement of each of the 5 plasticized filters, after a rotation of approximately 90°. Error bars represent the standard deviation.

- Concentrations obtained with instrumental relative sensibilities are slightly underestimated, showing that matrix correction is needed. The coefficient of determination shows good linearity.
- These samples are promising to be used as materials of reference for external calibration in XRF analysis under grazing incidence of PM filters [2, 3]. These data will support the development of the ISO/NP 23971 and VAMAS TWA 2 Project A34.

## WHAT'S NEXT?

- Samples will be measured also with other X-ray excitation sources, to detect the largest amount of elements.
- A wider range of concentration will be selected, to determine the upper and lower limit of linearity for each calibration curve.
- Samples will be digested and analysed with independent analytical techniques (ICP-MS) to determine absolute concentration and reproducibility.

### References

- EU, Directive 2008/50/EC on ambient air quality and cleaner air for Europe, Off. J. Eur. Union. L 152/1 (2008).
- P. Cirelli, F. Bilo, et al., *Assessment of calibration methods for Pb-loaded aerosol filters analysed with X-ray fluorescence under grazing incidence*, Spectrochim. Acta - Part B At. Spectrosc. 192 (2022)
- L. Borgese, F. Bilo, et al., *The assessment of a method for measurements and lead quantification in air particulate matter using total reflection X-ray fluorescence spectrometers*, Spectrochim. Acta - Part B At. Spectrosc. 167 (2020)

### Acknowledgment

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 COST Action CA18130 ENFORCE TXRF