

COMPARISON OF SYNCHROTRON-INDUCED X-RAY FLUORESCENCE WITH ED-XRF ON THE ELEMENTAL ANALYSIS OF AIR POLLUTION SAMPLES

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A common problem for elemental determinations of air pollution samples is the relatively low sample mass available for analysis. X-ray based techniques are desirable due to their non-destructive nature and cost-effectiveness compared to wet chemistry methods such as ICP-MS. However, the sensitivity of commercial ED-XRF systems tends to restrict air pollution samples to low time resolution (e.g. 24 hr) and large particle size bins (e.g. PM_{2.5} or all particles below 2.5 microns). In order to quantify air pollution samples with greater temporal and particle size information, higher photon flux may be beneficial.

In this study, the single compound and multi-elemental reference materials (ME-RMs) were generated using aerosol deposition system at the University of California-Davis (UCD)^{1,2}. Analyses were performed utilizing an ED-XRF (Panalytical Epsilon 5, Almelo, The Netherlands) operated at the University of California, Davis and quantified results were compared to the reference values.

In addition, selected RMs and air samples were analyzed using synchrotron-induced X-ray fluorescence (SXRF) operated at the Stanford Synchrotron Radiation Lightsource (SSRL). Beamline 2-2 at SSRL is a “white” light station providing an unfiltered flux of 10¹⁰ photons per second from a bending magnet with a declared energy range of 1 – 40 keV. The unaltered beam spot size is 4.0 mm x 8.0 mm but is typically trimmed to 0.4 mm x 3 mm to match the small sample deposit dimensions. The chamber used is capable of helium purging but is most often used in open air. Analysis time for this study was 20 seconds per analysis with each subject being analyzed at 5 different locations. The spectra were fitted using both the commercial WinAXIL program and the open-source PyMca program developed by the Software Group of the European Synchrotron Radiation Facility.

Selected ME-RM was analyzed at the International Atomic Energy Agency (IAEA) synchrotron (Elettra) facility in Trieste, Italy at the X-Ray Fluorescence beamline based on a bending magnet that gives a flux of around 5 · 10⁹ photons per second. A monochromatic beam with a size of 100 μm x 200 μm and an energy of 14 keV was used to scan a material area of 2.25 mm x 2.25 mm at a reflection geometry 45°/45° with a counting time of 300 s/pixel within a ultra-high vacuum environment at a pressure of 4 · 10⁻⁸ mbar. An X-ray detector was equipped with a super-light element window that enabled an efficient detection of low-Z elements.

The results of SXRF agreed well with reference values and EDXRF for few elements, including S, K, Cr, Fe, Cu and Zn. The results from the IAEA are comparable with the reference loadings and results obtained from EDXRF analysis for Na, S, K, Ca, Mn, Fe, Co, As, and Se.

[1] Indresand, H.; White, W. H.; Trzepla, K.; Perley, B. P.; Dillner, A. M., *X-ray Spectrom.*, 2012

[2] Yatkin, S.; Amin, H. S.; Trzepla, K.; Dillner, A. M., *Aerosol Sci & Technol.*, 2016