Evaluating XRF Na and Cl Measurements in Particulate Matter Samples

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The ratio of chlorine and sodium in a particulate matter (PM) sample can yield information about the origin or the chemistry of the collected particles. However, sodium is considered a difficult element to measure by ED-XRF due to its low cross section compared to other elements. Calibration of XRF systems for PM analysis is commonly done by using commercial NaCl and KCl standards on Mylar or nucleopore substrates. The commercial standards differ in potentially significant respects from PM collected on Teflon filters, and a calibration done by using standards that reproduce relevant characteristics of the sample and mass range can significantly improve the precision of the calibration factors and accuracy of the measurements (Indresand et al, 2011).

As a first approach to evaluate the Na and Cl measurement in a routine PM monitoring network, IMPROVE (Interagency Monitoring of PROtected Visual Environments, http://vista.cira.colostate.edu/IMPROVE/), the masses of Na and Cl on prepared NaCl reference materials were measured in two different ED-XRF systems; the IMPROVE custom-built Cu-anode system with a SiLi detector and a commercial system utilizing a CaF_2 target with a Ge detector. Reference materials were made by depositing pure and anhydrous NaCl particles onto 25 mm circular Teflon filters, exactly mimicking ambient PM samples. The mass range of NaCl was from 5 to 150 μg encompassing the IMPROVE network range for sites influenced by sea salt.

In the IMPROVE XRF system the Na and Cl XRF responses compared to the gravimetric mass of Na and Cl on each reference material were linear but different than unity. The Na response curve had a slope of 1.22 and an R^2 of 0.988 indicating that the IMPROVE XRF reported Na has a positive bias. The Cl response curve had a slope of 0.91 indicating a negative bias in the Cl measurement and an R^2 of 0.999, a better correlation and thus better precision than observed for the Na measurement. In the commercial system, the Na and Cl response lines were also linear with the same correlation coefficients (R^2) as in the IMPROVE system. However, both the Na and Cl responses were lower than unity at 0.92 and 0.96, respectively, indicating a negative bias in both measurements.

References

Indresand et al, Preparation of reference materials to evaluate and calibrate an x-ray fluorescence instrument for analysis of ambient particulate matter samples, X-ray spectrometry, 2011 (Submitted)