

Spectral Interferences of Sulfur on Light Elements in XRF Analysis of Particulate Matter Samples

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Quantitative XRF analysis is performed on atmospheric particulate matter (PM) samples to obtain masses of light elements Na, Mg, Al, and Si among others. Sodium, Mg, Al and Si are important elements in PM soil and sea salt source analysis, and secondarily for source control strategies, health effects studies, and for assessing visibility impairment and climate forcing caused by PM. In the US IMPROVE PM network (Interagency Monitoring of PROtected Visual Environments, <http://vista.cira.colostate.edu/IMPROVE/>) light elements are measured by a custom-built Cu-anode ED-XRF system with a SiLi drift detector. Empirical evidence from samples collected over several years and from over 150 sites across the US, show that Si is not reported correctly when samples are elevated in sulfur. Both an increase and decrease in Si has been observed. In XRF spectra of ammonium sulfate reference materials that mimic ambient PM samples a substantial low-energy tail has been observed on the sulfur peak that scaled with mass (Indresand et al, 2011).

The objective of this research is to demonstrate and quantify the spectral interference of sulfur on light elements in atmospheric particulate matter samples. We collected parallel ambient PM samples using IMPROVE filter (25 mm Teflon) samplers at a site giving a wide range of light element concentrations but low sulfur content. The samples were analyzed by the IMPROVE XRF system and by a commercial ED-XRF system with a CaF₂ target and a Ge detector. Synthetic ammonium sulfate particles were deposited in a layer onto the samples in the same manner as the samples are collected from the atmosphere. After addition of sulfur, the samples were re-analyzed by both XRF systems. The attenuation effect of light element x-rays from the sample through the layer of ammonium sulfate was modeled to determine if the layer was the cause of a potential signal decrease.

In collected samples with large light element masses, i.e. high soil particle content, the impact of sulfur in the IMPROVE XRF system was observed as an increase in Si mass by up to 40 % increasing with increasing S/Si ratios between 2 and 12 (ratios observed in network data). There was no change observed in control filters. The enhancement of Si is believed to be a function of the spectral interference of sulfur specific to the IMPROVE XRF system. In the commercial XRF system Si mass decreased down to 50 % of the original mass correlating with the S/Si ratios. This decrease of Si as a function of added sulfur in the commercial system was fully explained by the attenuation model.

The effect of sulfur on Na, Mg, Al, and Si for all samples in the whole range of light element masses will be described.

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)