PREPARATION OF REFERENCE MATERIALS OF LEAD (Pb) AND SILICON (Si) FOR XRF ANALYSIS OF AMBIENT PARTICULATE MATTER

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A disparity exists in elemental concentrations of commercially available XRF reference materials (RMs) and elemental concentration found in atmospheric particulate matter (PM) samples. The commercially available standards have different substrates (Nucleopore® or Mylar® as opposed to Teflon) and deposition pattern than atmospheric PM samples. In order to more closely mimic PM samples, we have made RMs for Pb and Si using an aerosol deposition method.

Pb is a highly toxic element in atmospheric PM. RMs of Pb were generated by aerosolizing an aqueous solution of lead acetate trihydrate. The generated particles were sampled on 47 mm Teflon filters (0.2 µm pore size, MTL, Minneapolis, MN, USA) using Thermo-Scientific 2025i Partisol Sequential Air Sampler (Franklin, MA, USA) with a flow rate of 16.7 L min⁻¹. For initial experiments, multiple filters at approximate Pb mass loadings of 0.2 µg/cm² and 0.6 µg/cm² (National Ambient Air Quality Standard for Pb is 0.15 µg/m³ or 0.3 µg/cm²), were generated. Twelve filters (six filters at each mass loading) were analyzed using two PANalytical Epsilon 5 (Westborough, MA, USA) energy dispersive (ED) XRF instruments calibrated with MICROMATTER™ (Vancouver, BC, Canada) standards. Each filter was analyzed three times on both instruments and relative standard deviation among the six reported values for Pb mass loadings was found to be lower than 4%. The Pb mass loadings were confirmed by an independent laboratory using Thermo-Scientific ARL QUANT’X EDXRF (West Palm Beach, FL, USA). The EDXRF results from two labs show less than 6% difference in mass loadings for eleven of twelve filters with an 8% difference for one of the 0.2 µg/cm² filters. Additional RMs with Pb mass loadings ranging from 0.1 µg/cm² to 1.2 µg/cm², on Teflon filters (MTL and Pall Life Sciences) will also be discussed.

The element Si is a major component of soil which can be a significant portion of particulate matter in the Western US. The Si loaded filters were generated using a suspension of 10 nm SiO₂ nanoparticles. The aerosolized suspension was collected on 25 mm Teflon filters (Teflo, 3.0 µm pore size, Pall Life Sciences, Ann Arbor, MI, USA) using Interagency Monitoring of Protected Visual Environments (IMPROVE) PM sampler. The mass loadings of Si were between 0.5 µg/cm² and 13.2 µg/cm². The range of Si concentration obtained from the non-urban network of PM sampling (IMPROVE: http://views.cira.colostate.edu/fed/Default.aspx) for 2011 ranges from 0.01 µg/cm² (5th percentile) to 3.9 µg/cm² (95th percentile). To determine if nanometer sized particles produce the same response as standards and PM samples, RMs will be generated using nanoparticles of Ti, an element that is well measured using EDXRF.