

**HEAVY METAL ANALYSIS OF WATER SAMPLES DOWN TO SUB-ppb  
LEVEL BY USING X-RAY FLUORESCENCE ANALYSIS COMBINED WITH  
THE CONCENTRATION TECHNIQUES**

Shinjiro Hayakawa, Masamichi Iwaki, Yuka Nishimoto, Kazuma Yamane  
and Takeshi Hirokawa<sup>a</sup>

*Department of Applied Chemistry, Graduate School of Engineering  
Hiroshima University, Hiroshima 739-8527, Japan*

Detection limit of x-ray fluorescence analysis has been greatly improved by using two types of concentration techniques. Both methods concentrate trace elements in the water samples onto a small piece of filter paper. One method utilizes a special sample holder that is made of a small filter paper (ca. 1mm<sup>2</sup>) attached onto a thin poly-ethylene (PE) sheet. A water droplet on the sample holder is concentrated onto the filter section because of the hydrophobic nature of PE. A water droplet of 1 mL can be processed within a day under ambient condition. The other method utilizes a small section of ion exchange cellulose paper (Whatman P-81). A suction filtration attachment made of Delrin has been developed, and cations in a water sample up to 9 mL can be concentrated onto the filter paper. A water sample of 1 mL can be processed in 3 minute.

The performance of these two methods are investigated with an x-ray fluorescence instrument composed of a sealed Mo tube, a graphite monochromator, beam collimator and a Si detector. The beam size on the sample is approximately 3 mm in diameter, and the x-ray tube is operated with 40 kV and 35 mA. The detection limit of the XRF instrument is less than 1 ng (Cu) in absolute amount. The detection limit for water samples is around 1 ppb level with the special holder, and the detection limit less than 1 ppb can be achieved with the ion exchange method. The sensitivity with the ion exchange method is strongly affected with the pH and the co-existing anions, and the quantification should be made with the standard addition method.

The principles of the developed methods, the feasibilities and their application especially for Pb analysis in the tap water and processed water will be presented.