

PPB ANALYSIS BY XRF; A NEW MICRO-DROPLET METHOD FOR ENVIRONMENTAL LIQUID SAMPLE

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X-ray fluorescence spectroscopy is used for environmental analysis because of its non-destructive, rapid, simple, and highly reproducible feature. Liquid samples such as sewage, waste water, industrial discharge, and river and sea water can be analyzed with XRF spectrometers by using liquid cells or a micro-droplet filter paper. A small amount of liquid is dropped on a filter paper and the dried paper with non-volatile elements is measured in the latter method. While liquid cells must be placed in He or other air with standard atmospheric pressure, the dried paper of the micro-droplet method can be measured in a vacuum and low Z elements such as Na, F, and B can be analyzed effectively. However, since the filter paper is now the matrix of the specimens to be measured, background scattering is increased causing the lower limit of detection (LLD) to deteriorate.

In this study, a new micro-droplet method “Ultra Carry” has been developed by using a new substance instead of the filter paper, and has reduced background scattering to improve LLD.

A standard solution (SPEX, LPC Standard1), which has 5-100 ppm of each element such as Pb, As, Cr, Se, Cd, etc, was analyzed by this new micro-droplet method. In this analysis, non-standard FP quantification program (SQX) was carried out with Rigaku ZSX spectrometers and good results were obtained. The LLD achieved with this method was sub ppm for several elements (e.g. Na 76ppb, P 56ppb, K 12ppb, Cr 26ppb, As 16ppb, Se 24ppb, Cd 182ppb, Pb 76ppb), 1ppm for F and 30ppm for B.

Calibration lines for Pb, As, Cr, Se, and Cd were generated with standard solutions of river water. In this study, 50mL sample was concentrated to about 1mL with a rotary evaporator, and the concentrated sample was dropped twice by 500 μ L on the new substance. The reproducibility tests (n=5) were performed, and standard deviation of a few dozen ppb was obtained for each element (Cr 17ppb, As 14ppb, Se 27ppb, Cd 29ppb, Pb 24ppb).

The newly-developed micro-droplet method, which is easy, simple, and highly reproducible, enables to analyze ppb of trace elements and ppm for F and B in water with a standard XRF spectrometer.