

# Energy-dispersive X-Ray fluorescence spectrometry as progressive technique for the determination of elements in stream sediments

Augustín Majchrák\*, L'udmila Pencáková\*

## Intoduction

In most environmental labs inorganic analysis is divided into two areas — ICP analysis and AAS analysis. In this paper we will focus on the third possibility exploration of energy-dispersive X-ray fluorescence spectrometry for the determination of elements in stream sediments. Stream sediment is a material which represents the accumulation of distributed elements from anthropological activities and mineral deposits. XRF instrument can be used to quantify most of the elements in the periodic table over a wide dynamic range. This technique was explored to this purpose.

Energy-dispersive X-ray fluorescence analysis (ED XRF) is a non-destructive analytical technique for the determination of chemical elements in solid, powders and liquids. The advantage of ED XRF include simple samle preparation, high reliability and excellent reproducibility of analytical results. Its physical basis is the use of X-rays to excite the elements in the material being tested, causing them to emit fluorescence radiation. Measuring the energy and intensity of these emissions allows the elements to be identified and quantified. ED XRF analysis is ideal method for

determination of samples with variable composition, because all elements from atomic number  $Z = 11$  (Na) to  $Z = 92$  (U) are analysed simultaneously in whole concentration range and samples preparation is very simple. Polarised X-ray radiation significantly improves the detection limits of energy-dispersive X-ray spectrometry.

## Experimental

ED XRF instrumental conditions were optimised for the determination of K, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Nb, Sn, Sb, Cs, Ba, Pb in stream sediments. Accuracy was evaluated by analysis of certified reference materials for stream sediments from USA, China, Canada, Slovakia etc.

All measurements were performed on the SPECTRO X-Lab 2000 ED XRF spectrometer. The SPECTRO makes use of different analytical models using fluorescence lines and scattered radiation. Spectral processing is an automated and routine operation, the program selects the optimum evaluation procedures. An interactive processing procedure allows to influence the various evaluation steps and select their individually optimised parameters. For determination elements we used method prepared for geological materials by firm SPECTRO. To achieve more accurate results we modified this method and enhanced number of reference materials of stream sediments.

---

\*Geological Survey of the Slovak Republic, Geoanalytical Laboratories, Markušovská cesta 1, 052 40 Spišská Nová Ves, Slovakia

We compared this method with AAS and AES-ICP methods and result were in good agreement. The precision of the measurement ranged from 1 to 3% RSD. We can say that this method is comparable with the other methods for determining elements in stream sediments.

Results of analyses for some elements in Buffalo river sediment by ED XRF method are presented in tab. 1.

### **Conclusion**

By comparison, with AES-ICP, AAS methods, non-destructive method X-ray energy-dispersive fluorescence spectrometry has, for some selected elements, the same detection limits, reliability and reproducibility as well. We can say that X-ray is less energy, material and time consu-

**Tab. 1**

<b>Element</b>	<b>ED XRF value [<math>\mu\text{g}\cdot\text{g}^{-1}</math>]</b>	<b>Certified value [<math>\mu\text{g}\cdot\text{g}^{-1}</math>]</b>
Cr	134	135 $\pm$ 5
V	94	95 $\pm$ 4
Sn	9.2	9.5 $\pm$ 0.5
Sb	3.9	3.79 $\pm$ 0.15
Cu	96.5	98.6 $\pm$ 5.0
Zn	437	438 $\pm$ 12
Ba	426	414 $\pm$ 12

ming method to destructive methods and thus is more preferable.