Application Note: 41635

Analysis of Soils and Stream Sediments

ARL ADVANT'X Series with IntelliPower™
Sequential X-Ray Fluorescence Spectrometer

Key Words

- ARL ADVANT'X - 3600W
- Soils and Sediments
- XRF
- X-Ray Fluorescence

Introduction

This application note presents analytical results obtained from a series of river sediments, soils and stream sediment samples using the Thermo Scientific ARL ADVANT'X Series spectrometer.

The main objective of this study is to obtain the typical limits of detection for a broad range of toxic and other trace elements in sediments and soils. Although both solid and liquid samples can be analyzed with the ARL ADVANT'X instrument, only solid samples are analyzed in this study.

The ARL ADVANT'X instrument is equipped with a universal goniometer that covers most of the elements from beryllium to uranium provided the correct crystals are fitted. Flexibility and sensitivity are two important prerequisites for the analysis of trace elements in the geological samples such as sediments.

In order to achieve the best possible limits of detection for trace elements in soils and stream sediments, optimal excitation conditions, accurate background and matrix correction procedures are essential.

The WinXRF software package allows background corrections employing a wide variety of models, spectral line overlap correction by different procedures and matrix correction using the Rhodium Compton peak intensity as internal standard. The high performance generator also provides optimal excitation of all elements measurable by X-ray fluorescence.

Samples and methods

A series of 8 certified reference materials (soils and sediments) were chosen for the analysis. The powders were pressed into pellets without any binding agent. Background corrected net intensities were used to set up calibration curves for all the elements of interest. X-ray tube conditions, collimator-crystal-detector choice and counting time were optimized to obtain the highest sensitivities.

Results and discussion

Figure 1 shows the XRF scan of two samples as an example: a soil and a sediment around Pb Lβ line used for the analysis of lead. One can also see the presence of other elements like As and Rb in the same scan. The background positions (left and right) are also indicated in the figure.

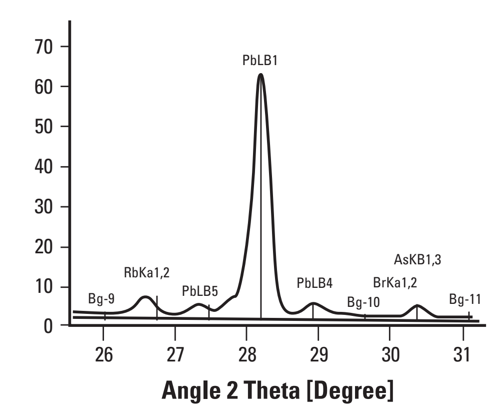

Figure 1: XRF scan of a soil and a sediment around Pb Lβ line used for the analysis of lead.

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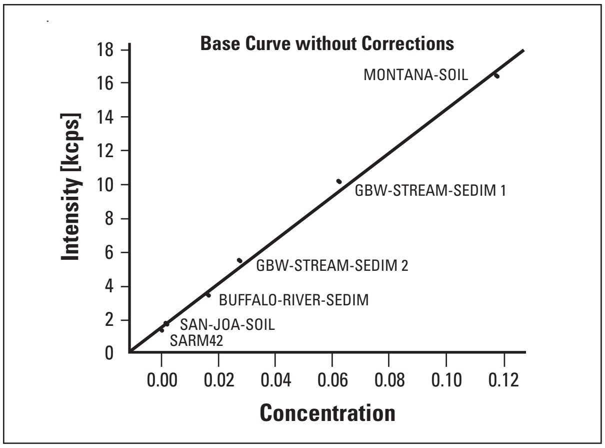

Figure 2: Example of a calibration curve obtained for Pb

Figure 2 shows an example of a calibration curve obtained for Pb. A standard error of estimate (accuracy) of 0.0012 % is obtained. A limit of detection of 1.1 ppm is achieved for this element. Similar calibration curves were prepared for other elements as well. The results are shown in Table 1 which includes the limits of detection (expressed as 3σ, 100s counting time) for all the elements measured in the soil and sediment samples.

ELEMENT	RANGE [PPM]	SEE [PPM]	LOD [100S] 3 σ	PRECISION IN 100s [PPM]	AT THESE CONC [PPM]
As	17.7 - 626	15.9	1.8	±2	at 340
Cd	0.1 - 42	5.3	2.5	±1.5	at 40
Co	8.5 - 35	2.8	1	±1	at 9
Cr	345 - 4310	6.6	0.7	±1.6	at 44
Cu	17 - 2950	4.6	0.6	±4	at 2800
Hg	0.1 - 33	3	1.3	±1.9	at 30
Mo	1.3 - 19	1.6	0.3	±0.1	at 20
Ni	13 - 88	2.6	0.6	±0.2	at 10
Pb	10 - 1162	12.3	1.1	±10	at 1000
Sn	10 - 370	1*	3.2	±1.6	at 25
V	46 - 170	6.5	0.3	±0.5	at 74
Zn	44 - 498	13.8	0.3	±2.8	at 320

Table 1 : Typical limits of detection in soils and sediments using the ARL ADVANT'X instrument

SEE: standard error of estimate
LOD: limit of detection (3 sigma)
Precision: repeatability obtained on 10 repeat measurements

*Only 3 samples

UniQuant® standardless analysis

Finally, one of the samples was analyzed using the UniQuant® program which is capable of determining the concentration of most of the elements present in any "unknown" or "non-routine" sample without standards or calibration curves. This program measures XRF intensities at various spectral positions followed by data treatment to obtain the concentrations. Table 2 shows the UniQuant® results compared to the certified values for various elements in a stream sediment sample.

ELEMENT	CERTIFIED	UNIQUANT®
Pb	636 ppm	590 ppm
Rb	408 ppm	410 ppm
Zn	373 ppm	300 ppm
Sn	370 ppm	229 ppm
Cl	290 ppm	350 ppm
P	260 ppm	310 ppm
As	188 ppm	180 ppm
Zr	153 ppm	140 ppm
W	126 ppm	160 ppm
Cu	78 ppm	70 ppm
Ce	58 ppm	38 ppm
Bi	50 ppm	45 ppm
V	47 ppm	40 ppm
Y	43 ppm	43 ppm
Cr	40 ppm	35 ppm
Sr	29 ppm	28 ppm
Nb	25 ppm	24 ppm
Ni	14 ppm	13 ppm
U	9 ppm	10 ppm
Er	4.6 ppm	4.9 ppm

OXYDE	CERTIFIED	UNIQUANT®
SiO_2	76.2 %	75.61 %
Al_2O_3	10.4 %	11.52 %
Fe_2O_3	4.4 %	4.01 %
K_2O	3.28 %	3.24 %
MgO	0.62 %	0.67 %
CaO	0.47 %	0.43 %

Table 2 : UniQuant® results compared to the certified concentrations for various elements in a stream sediment sample

Conclusion

Thanks to the high sensitivity and analytical flexibility offered by the ARL ADVANT'X spectrometer, both quantitative and semi-quantitative analysis can be performed without any compromise down to sub-ppm levels in stream sediments and soils. Very high reproducibility can be obtained even for elements with concentration levels below 10 ppm.

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