On the use of a High-Purity Ge-Detector for PIXE Spectrometry on Geological Materials.

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Abstract

A comparison between a conventional Li-drifted Si-detector and a high-purity Ge-detector is made by performing PIXE analyses on the geological reference material MP-1 with a nuclear microprobe. Measurements were made during a routine type of run; no special circumstances were created during this work.

Introduction

For the majority of work done in the field of X-ray spectrometry, Li-drifted Si-detectors are used. Independent of the way the X-rays are generated, this detector offers adequate energy resolution and efficiency characteristics for most applications. Especially for conventional energy dispersive XRF, the low efficiency beyond about 20 keV is an advantage as the detector has a low response for the exciting radiation.

Excitation with charged particles (electrons, ions) however, may benefit greatly from the higher efficiency at high energies of Ge-detectors, as no hard X-ray background is present. Moreover, the better peak to background ratio enables more precise measurements of low Z elements in a heavy matrix. In the case of PIXE, simultaneously with the X-rays low energy γ-transitions can be measured for the analysis of light elements, in this way avoiding complicated multi-detector calibration procedures. These advantages outweigh the appearance of Ge-escape peaks and the discontinuity in the efficiency curve due to the Ge absorption K-edge.

Experimental

For the comparison mentioned above, the Amsterdam nuclear microprobe was used. The proton energy was 3.0 MeV, a 100 pA beam current was used, focused into a beam spot of less than 5 μm. The beam current was adjusted in such a way that the counting rate for the Ge-detector did not exceed 5 kHz.

The characteristics of the 2 detectors used are summarized in Table 1.

Table 1.

	Si(Li)-detector	*Ge-detector*
type	SLP-04165-S	IGLET-X-11145-S
active diameter (mm)	4	11
active depth (mm)	3.27	10
Be-window (μm)	25.4	25.4
resolution at 5.9 keV (eV)	165	145

In order to absorb very intense low energy X-rays, an extra 50 μm Al-absorber was installed in the path between sample and detectors. Detector signals were fed into an ORTEC 671 main amplifier and an ORTEC CAMAC-based ADC; the digitized data were read out by a VME bus coupled to a SUN work station on which a home made data-acquisition program runs.

In Table 2 results of the analyses of the reference material are given. This reference material consists of an ore from the deposit of the Brunswick Tin Mines Limited in South-Western New Brunswick. It is basically material from two sulphide veins blended with a small amount of mineralized rock and a good representative for geological materials. In the table, reference values are also given. It should be noted that deviations from these values are largely due to inhomogeneities in the reference material on a micro-meter scale. With a microprobe of this dimension, different spots will result in different concentration values. For the comparison of the detectors, however, exactly the same spot has been irradiated with the proton beam.

The X-ray spectra (given in fig. 1 and 2) are evaluated with the computer code GUPIX, developed at Guelph-University (Guelph, Canada); this code is commercially available. The code calculates concentrations taking into account matrix effects known in PIXE such as the slowing down process of the protons with subsequent decrease of the inner-shell ionization cross sections; absorption of X-rays on their way through the sample towards the detector, pile-up processes and secondary fluorescence. The code has available efficiency curves for both types of detectors.

Table 2.

Element	*Reference value*	*Ge-detector*	*Si-detector*
Ca	3.4%	3.3% (5.3)	3.3% (15)
Ti	0.07%	160 ppm (66)	N.D.
Cr	-	127 ppm (21)	N.D.
Mn	0.05%	589 ppm (5.0)	N.D.
Fe	5.7%	3.7% (0.3)	3.4% (1.1)
Ni	-	330 ppm (2.7)	315 ppm (29)
Cu	2.1%	1.6% (0.4)	1.4% (1.7)
Zn	15.9%	14.1% (0.2)	13.9% (0.6)
As	0.8%	0.78% (1.9)	0.70% (5.2)
Cd	0.07%	837 ppm (11)	N.D.
In	0.07%	975 ppm (15)	N.D.
Sn	2.4%	2.3% (2.3)	1.8% (12)
Pb	1.9%	1.6% (2.0)	1.1% (9.2)

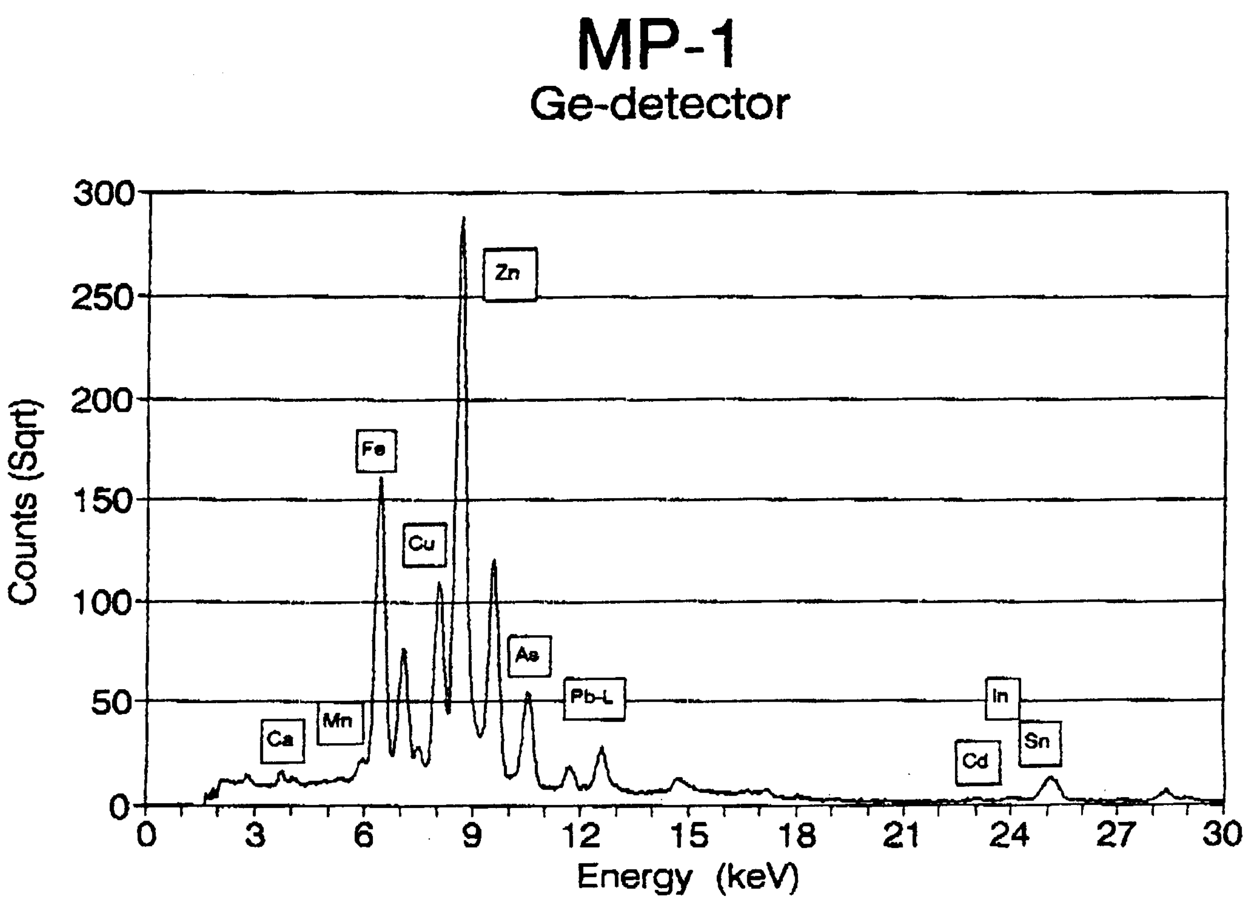
The figures in brackets indicate the percentile error in the results as given by the fitting procedure. N.D. stands for not detectable.

Conclusion

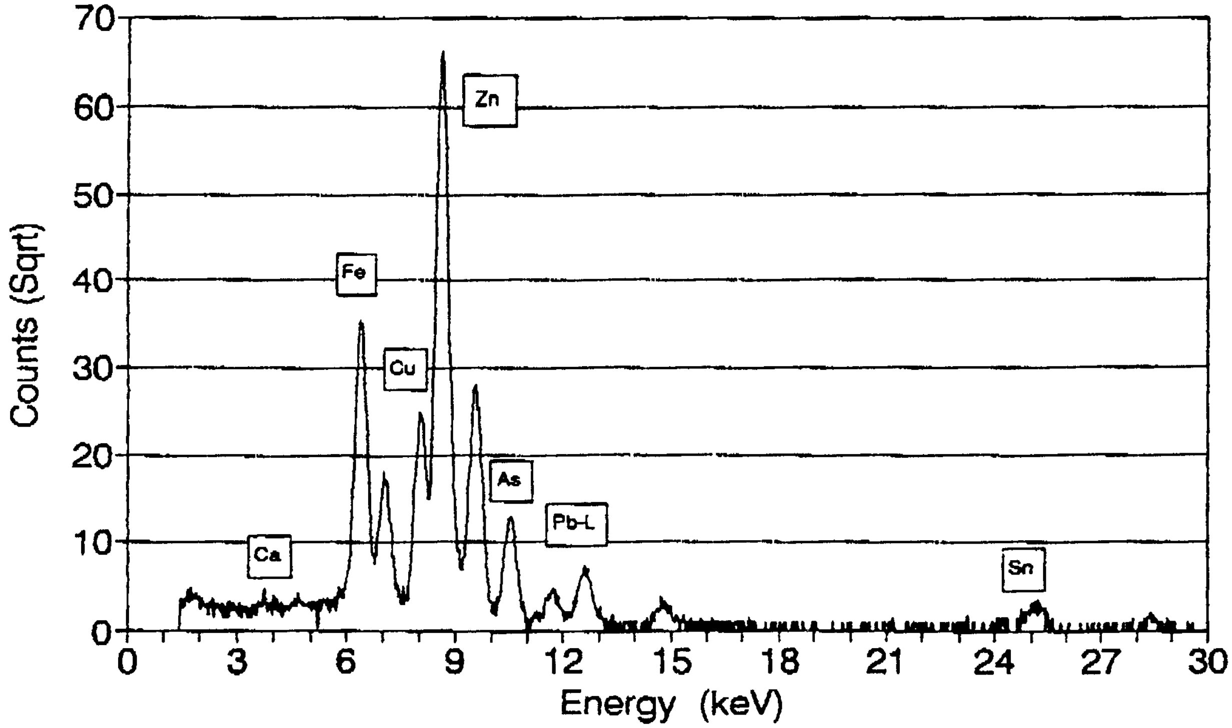
By looking at both the table and the figures of the spectra, one can draw the following conclusions:
— With the Ge-detector more elements can be detected. This is largely due to the larger solid angle. Longer irradiation times with the use of a Si(Li) detector will obtain comparable results, but one should realize that especially during microprobe work, with the aim being to measure concentration maps in reasonable measuring times, this will inevitably lead to small dwell time of the beam on a pixel. A large solid angle with high efficiency is a must also because of the fact that not all samples can withstand long irradiation on the same spot. Moreover, Si-detectors of comparable dimensions will at the present stage of development suffer from a worse energy resolution.
— Again due to the much better statistics, the errors in the results of the Ge-detector are substantially smaller.
— High energy peaks can be measured with the Ge-detector. In this example it turned out to be very helpful to have an indication of the K-X-rays of Pb. The well-known spectral interference between Pb-L-X-rays and As-K-X-rays can be tackled much more convincingly if this extra information is available.

— The Ge-detector offers the possibility to measure nuclear transition, as can be seen in fig. 2, where F is visible. No quantification of the element has been performed, but the possibility is present provided that a suitable standard is available.
— The Ge-detector has definitely an elevated background between about 11 and 18 keV (fig. 1) due to pile up. Careful choice of the beam current, absorbing material and, preferably, a beam on demand system that turns the beam off during pulse processing can improve the situation.

As an overall conclusion the Ge-detector is a very attractive option for the collection of X-rays emitted after excitation with charged particles. It is especially the good ratio between size of the crystal and energy resolution in combination with the current limitations of charged particle beams that makes this type of detector attractive.



Si-detector



MP-1

Ge-detector