MULTI-ELEMENT STANDARD FOR ROUTINE INSTRUMENTAL ACTIVATION ANALYSIS OF TRACE ELEMENTS IN ROCKS AND TECTITES

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The use of high-resolution Ge(Li) detectors in non-destructive instrumental neutron activation analysis (INAA) of mineral materials makes possible the simultaneous determination of a number of trace elements. In routine applications of INAA the use of a multi-element standard (MES) has proved to have advantages over a set of standards for each determined element. An MES has been prepared containing 8 trace elements mixed in a suitable proportion and giving, after neutron activation, long-lived $\gamma$-emitters, the $\gamma$-ray lines of which regularly occur in the $\gamma$-ray spectra of silicate mineral materials. This method has been used in the determination of Sc, Cr, Co, Rb, Cs, Eu, Hf and Th in samples of standard rocks and moldavites.

Introduction

The analysis of mineral materials is one of the most exacting tasks in analytical chemistry, because of the complex composition of the sample and the difficulties connected with the decomposition. As an instrumental method, non-destructive neutron activation analysis followed by $\gamma$-ray spectrometry considerably simplifies and shortens the analytical procedure. The high resolution of the semiconductor Ge(Li) $\gamma$-ray detectors (about twenty times better than that of scintillation detectors) brings higher selectivity, resulting in a wider range of potential uses for instrumental neutron activation analyses by allowing simultaneous determination of a great number of elements present in a wide variety of combinations. Quantitative determinations of individual elements are carried out essentially by an absolute method, the single comparator method, or by a relative method.

The absolute method in activation analysis is used only in isolated cases.\(^1\) The reason for this is mainly the problem of determining the absolute activity and the accurate value of the neutron flux; in a large number of cases the inaccuracies in the values of nuclear constants necessary for the calculation (cross-section, half-life) are of great inconvenience.

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The single comparator method is based on simultaneous irradiation of samples and the comparator, i.e. a standard amount of an element which gives a suitable radionuclide (e.g. $^{60}$Co) by activation. The selected comparator is activated together with the standards and their spectra are filed. This allows us to compare at any later time the intensities of the $\gamma$-rays of the activated samples with the intensities of the filed $\gamma$-transitions in the spectra of the standards. The degree of activation is compared by means of the $\gamma$-ray intensities of the comparators from both activations. This method requires a time-independent energy spectrum of the activating neutrons and a time-independent energetic efficiency of the detector. It is very suitable for the determination of many elements in one sample, for the analysis of a great number of samples.

The relative method, together with all its modifications, is the most frequently used procedure in activation analysis. The fact that in a multi-component analysis the standards of all the determined elements must be irradiated simultaneously with the sample is the disadvantage of this method. A considerable part of the volume of the irradiation can is blocked by the standards, and the measuring of all individual standards lengthens the measuring time allotted for the analysis per sample.

In addition to sensitivity and selectivity, the cost per sample analyzed is yet another decisive factor in favour of routine application of the non-destructive activation method. Since with this method the cost per sample analyzed is dictated not only by activation but also by the time taken up in measuring, it is desirable to shorten the measuring time both of the sample and the respective standards as much as possible. A suitable solution has been found in modifying the relative method by using a multi-element standard (MES), which is a mixture of standards of the elements being determined. Solid materials (alloys, samples of rocks) can also be used as MES. Their composition must be analogous to that of the examined sample, with exactly known contents of the elements to be determined. It is preferable to use an artificially mixed solution, from which known volumes are withdrawn by micropipette. The optimum requirements for a suitable MES are as follows:

1. The concentration of individual elements should be such as to allow the line intensities of the determined $\gamma$-transitions to be mutually comparable and well measurable after irradiation. The integral activity of the MES must be optimum with respect to the length of measuring and the dead-time, and should be comparable with the anticipated integral activity of the samples to be measured together with the MES.

2. The half-life values of the radionuclides arising ought to be comparable to permit evaluation from one measurement. However, it is not ruled out that MES be composed of elements whose activation gives rise to two or more groups of radionuclides, according to the value of the half-life.

3. Intense $\gamma$-transitions which can be used for quantitative evaluation of the target element abundances must be well distinguishable.

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