RAPID AND SIMULTANEOUS DETERMINATION OF U, F, Al, Ca AND V IN PHOSPHATE ROCKS BY A COMBINATION OF DELAYED NEUTRON AND γ-RAY SPECTROMETRY TECHNIQUES

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Delayed neutron counting for uranium assay was coupled with γ-ray spectrometry in order to measure radionuclides of F, Al, Ca and V produced by neutron activation. For this measurement, the delay time required for U determination was exploited. Calibration of the method was provided by standards based on a CaCO₃ matrix and validity of results was verified against other analytical methods. A single sample can be analyzed for the five elements in 3 min with a precision of ~±10%. The method developed was applied in the exploration of phosphate ores. The measured total concentrations permitted the disclosure of correlations between various elements and constituents in the samples. Chemical and mineralogical properties were obtained as well.

Introduction

Neutron activation analysis followed by delayed neutron counting is a well known method for sensitive and accurate determination of uranium /1/. This method was recently explained in detail by S. Amiel /2/

In reactor neutron activation most delayed neutron precursors are fission products of ²³⁵U. These delayed neutrons from fission are accompanied by delayed neutrons emitted by ¹⁷N (T₁/₂ = 4.17 s). In order to eliminate this interference from ¹⁷N, a delay time of about 20 seconds after the end of irradiation is required before counting is started. This delay is also needed due to the experimental error resulting from the transit time in the rabbit transport system (~2 s).

The longest half-life of delayed neutron precursors is about 1 min and therefore a fast and accurate transport system for sample irradiation is needed. In the last 10 years many transport systems for the activation analysis technique have appeared /3-6/.

The Flexo-Rabbit Pneumatic Transfer System, which provides simple and rapid transportation of the sample, has been installed in many laboratories. Delayed neutron counting is also used in many laboratories in uranium exploration /7/. However, none of these laboratories exploited the delay time required for U analysis for simultaneous determination of other elements based on γ-ray spectrometry.

The aim of this study was to combine the well known delayed neutron counting technique with γ-ray spectrometry and to exploit the delay time required for U determination for the simultaneous determination of F, Al,
Ca and V. The present method was applied in phosphate exploration in Israel in order to characterize the chemical and mineralogical composition of the ores.

Experimental

Irradiation

The irradiations were carried out in the Flexo-Rabbit Pneumatic Transfer System (Reactor Experiments Inc., CA, U.S.A.) of the IRR-1 reactor (see Fig. 1). Modular components of this system were incorporated so that the counting end for γ-spectrometry was also used as a delay station for delayed neutron counting. The advantage of such a set-up is two-fold:

a) During the delay period of a sample for U determination, γ-spectrometry measurements are performed.

b) The life span of the BF₃ counters is prolonged since they are not exposed to the delayed neutrons from the short-lived ²³⁵U groups.

All samples and standards were irradiated for 20 s in a neutron flux of $5 \times 10^{12}$ n/cm². s.

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Fig. 1. Flexo-rabbit pneumatic transfer system for irradiation, γ-ray spectrometry and delayed neutron counting; V₁0 = load, V₁1 = fire, LS₁₀ = escapement close, LS₁₁ = capsule in place, ◦ electrical connector, capsule photodetector, △ air connection 2.5 atm., ◇ air connection 7 atm., ○ air connection 1 atm.