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SHORT-TIME ACTIVATION ANALYSIS FOR GEOLOGICAL SAMPLES BY USING LOSS-FREE COUNTING

K. M. OCHSENKÜHN[1], N. N. PAPADOPOULOS[1], N. D. ANTONIOU[1] & N. KARISTINNEOS[2]

ABSTRACT

In geological research multielement analysis of a large number of samples is usually required. By introducing the loss-free counting technique into neutron- activation analysis, which permits gamma-ray spectrometry at high-count rates and dead times with instrumental count loss correction, the application range for a series of elements can be broadened. This technique allows the reduction of the waiting and counting time, shortening in that way the overall time of analysis and increasing the sample throughput.

KEY WORDS: short-time activation analysis, loss-free counting, high-rate gamma spectrometry

1. INTRODUCTION

As for geological explorations often a lot of different samples have to be collected, it is necessary to, have a good throughput of samples for the analysis. The evaluation of the trace element content in geological samples can give information about the genesis and the chemical environment of rocks or orebodies. Neutron activation analysis is up to now a choice for the analysis of a series of trace elements. A fast analysis (irradiation, cooling and measuring time) was, however, often, hindered by the nonexpected high radioactivity of some samples, which could therefore not be measured and which forced the analyst to prolong the cooling time, with the result that for those samples the whole spectrum of gamma-lines changed in comparison to the other samples, making the later obtained spectra not comparable to the earlier measured ones. By the use of loss-free counting technology (LFC), introduced by Westphal (1985), also samples with higher radioactivity can be measured besides the less radioactive samples (OCHSENKÜHN et al 1996).

The aim of this study was therefore to use LFC technology in connection with epithermal irradiation for a fast analysis of geological samples and to show which elements can be analyzed under these circumstances.

2. EXPERIMENTAL

Samples and standards . For the gamma spectroscopy of geological samples after epithermal irradiation, lignites and volcanic ashes were investigated. The samples were pre-crushed, then dried by using freeze-drying technique and finally powdered in an agate mortar. As reference materials the standards SO-2, SY-2, SY-3 and BX-N of the ARNT (Association Nationale de la Recherche Technique) were used. About 50 mg of the powders of the samples, exactly weighted, were transferred into polyethylene-containers (8mm id. and 0.3 mL vol.). Always four of the sample containers were collected in larger poly-ethylene containers [10mm id. and 5 mL vol.], which were heat-sealed. Furthermore for the irradiation a series of 9 samples 3 standard reference materials of about 50 to 100 mg each were used. The above described containers were placed into a special Cd container, which was subjected to neutron

[1] Dr. NCSR "Demokritos", Aghia Sophia, 15310 Athens, GREECE
[2] Dr. Institute for Geological and Mineral Exploration, Messogion Str. 70, 152 37 Athens, GREECE

irradiation. The Cd-tube has a basis and three wings to keep the samples in a optimum irradiation position.

Irradiation and measurement. The Cd-container with the samples was irradiated at the 5 MW swimming pool reactor of the NCSR "Demokritos", Athens, for 30 minutes at a thermal neutron flux of 2 x 10^13 neutrons/ cm^2 x sec. After a cooling time of 24 hours the Cd-container was opened under water, the samples moved- to the surface and were collected over there. The sealed containers were opened, the sample containers were transferred without opening into plastic-tubes and were immediately ready for measurement. The counting time was 15 minutes on a 37 cm^2 Ortec Ge(Li) semiconductor (FWHM = 2.17 keV for the Co-60 photopeak at 1332 keV, efficiency=15 % related to a 3"x 3" NaI(Tl) detector) (Canberra Industries, USA). The gammaspectra were obtained by using a loss-free counting unit in connection with the Accuspec 4096 channel multichannel analyzer system (Nuclear Data).

Data evaluation : The gamma-spectra of the samples and standards were evaluated at a IBM-PS/2 computer by the program PC/SPAN V 3.0, software package of the I.A.E.A. (International Atomic Energy. Agency), using for the peak area and peak error determination a Gaussian fitting method. For quantitative determination, the relative method was used. Although some samples had higher radioactivity with deadtimes much more than 20%, loss-free counting made it possible to measure all samples without any problem. The evaluation of the spectra showed the presence of gamma lines of the isotopes As76, Eu-152m, Ga-72, Gd-153/Sm-153, La-140, Np-239, Mn-56, K-42 and Na-24. Furthermore the isotopes Br-82, Ce-143, Ce144, Fe-59, Gd-159, Ho-166, Pa-233, Pm-149, Sc-47, Sm-153 and Zn-69m could be found in different samples.

Table 1. Results of the analysis of lignites and volcanic ashes by gamma-spectroscopy after epithermal irradiation

Element								
	As	Eu	Ga	La	U	K_2O	Mn0	Na_20
			[µg/g]				[%]	
Sample								
lignite(1)	10.7	0.59	7.4	16.6	22.7	0.55	0.014	0.3
lignite(2)	5.51	0.84	16.8	26.0	5.0	1.26	0.02	3.74
lignite(3)	5.94	0.31	3.35	7.75	9.2	0.19	0.019	0.087
volc.ash(1)	15.7	0.55	15.5	33.7	1.3	2.38	0.074	4.67
volc.ash(2)	13.3	0.69	14.6	24.1	1.36	1.64	0.092	3.54
volc.ash(3)	5.91	0.63	16.0	30.4	4.03	2.61	0.10	5.09

3. RESULTS AND DISCUSSION

Loss-free counting based on instrumental count loss correction at high, even variable, count rates and corresponding dead times allows to reduce the waiting time of the analysis of geological samples by neutron activation analysis and-increases in that way the sample throughput. It has also the advantage that samples with large element concentration and nuclide half-live range can be measured without any spectrum distortion. even if the count rate and dead time of some sample measurements is high. While the. lignites in Tab.l had deadtimes of about 5-10 % and could have been measured also with the conventional counting technique, the vulcanic ashes showed deadtimes between 25 and 35 % and therefore loss-free counting was necessary for their measurement.

This analytical procedure allows the determination of some elements with high sensitivity (OCHSENKÜHN et al 1995). The main purpose is the use of isotopes with half-lives of some hours to some days. That is the reason, why especially the elements As (As-76, $T_{1/2}$=1.096 d), Eu (Eu-152m, $T_{1/2}$=0.389 d), Ga (Ga-72, $T_{1/2}$=0.6 d), La (La-140, $T_{1/2}$=1.678 d) and also Mn (Mn-56, $T_{1/2}$=0.1075 d) arised in the gamma-spectra. However the analyst has to face the possibility to measure samples of higher

radioactivity, with the fact that the background of the gamma-spectrum is higher and, therefore, also the deviation of the background increases. Thus higher count rates of the isotopes of interest are necessary for the recognition of the respective peaks. The optimization of measuring on one hand in one runs as much isotopes as possible and on the other hand to obtain the best peak to background ratio is therefore the main problem of the analyst.

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