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DETERMINATION OF FLUORINE IN PHOSPHATE ROCKS BY 14 MeV NEUTRON ACTIVATION ANALYSIS

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ABSTRACT

Elemental fluorine in phosphate rocks has been determined using 14 MeV neutron activation and employing the reaction $F^{19}(n,p)O^{19}$. The gamma ray spectra were taken by a high resolution Ge(Li) detector and a fast pneumatic system was used to transfer the irradiated samples.

ÖZET

Fosfat cevherlerindeki fluor miktarı hızlı nötron aktivasyon analizi yardımıyla tayin edilmiştir. Bu tayinde $F^{19}(n,p)O^{19}$ reaksiyonundan yararlanılmış ve gama spektrumlarının alınmasında ayırma gücü yüksek Ge(Li) dedektörü kullanılmıştır. Işınlanan nümuneler, hava basıncıyla çalışan bir pnömatik sistem yardımıyla transfer edilmiştir.

INTRODUCTION

Phosphate rocks are the raw material of phosphoric acid, super phosphate, phosphor, and other phosphor compounds, and contain fluorine. During the fabrication of phosphate rocks fluorine is released as hydrofluoric acid. If it is not isolated and used as a by - product it may cause some difficulties because of its corrosive effect on glass, metal, and porcelain as well as its poisonous effect [1, 2]. Therefore, a limited amount of fluorine in the phosphate rocks is required to avoid any difficulty during fabrication.

The quantitative analysis of fluorine by neutron activation analysis is more rapid than conventional methods, the former is clearly a better choice from the standpoint of being repetitive and non - destructive.

Four separate thermal and fast neutron - induced reactions can be used for fluorine determination (Table — 1). Among the fast neutron - induced reactions, $F^{19}(n,p)O^{19}$ reaction is chosen to eliminate the interference of competitive reactions produced by other elements in rock samples. The short half - life of O^{19} necessitates the use of a fast transfer system. Also a high resolution gamma ray spectrometer is required

to isolate the 0.2 MeV gamma-ray full energy peak which is accepted for activity evaluation.

The ground samples were provided from the Mining Research and Exploration Institute of Turkey (MTA), and NaF was used as standard for comparative analysis.

TABLE — 1. The neutron reactions of fluorine [3].

Reaction	Cross section for $E_n = 14$ MeV (mb)	Product	
		Half-life	Gamma energy (MeV)
$F^{19}(n,\gamma)\ F^{20}$	9.8 ± 0.7 (Thermal)	11.2 sec	1.63
$F^{19}(n,2n)\ F^{18}$	47 ± 4	109.7 min	0.51
$F^{19}(n,p)\ O^{19}$	20 ± 2	27 sec	0.2;1.37
$F^{19}(n,\alpha)\ N^{16}$	15 ± 5	7.1 sec	5-7

EXPERIMENT AND RESULTS

The experimental set-up to determine fluorine consists of a neutron generator, a pneumatic fast transfer system which have been described elsewhere [4], and a gamma-ray spectrometer consisting of 60 cc Ortec Ge(Li) detector with a resolution of 3 keV, Canberra-8100 multichannel PHA, and related electronics.

The powdered samples and NaF standard were irradiated in suitable polyethylene capsules. Prior to analysis, pure CaF_2 was used for fluorine determination. The calculated and experimentally determined fluorine amounts in CaF_2 was found in good agreement within the error limits. The gamma-ray spectra of standard NaF and an apatite sample were given in Figs. 1, 2. The background and dead-time corrected full energy peak areas were calculated according to Covell method.

The oxygen isotope, O^{16} can give rise to N^{16} activity by (n,p) reaction. A suitable cooling time was chosen so that a possible interference due to the compton continua from high energy gamma-rays of N^{16} could be avoided.

Results of the fluorine analysis with their errors derived from the repeated measurements were given in Table — 2.

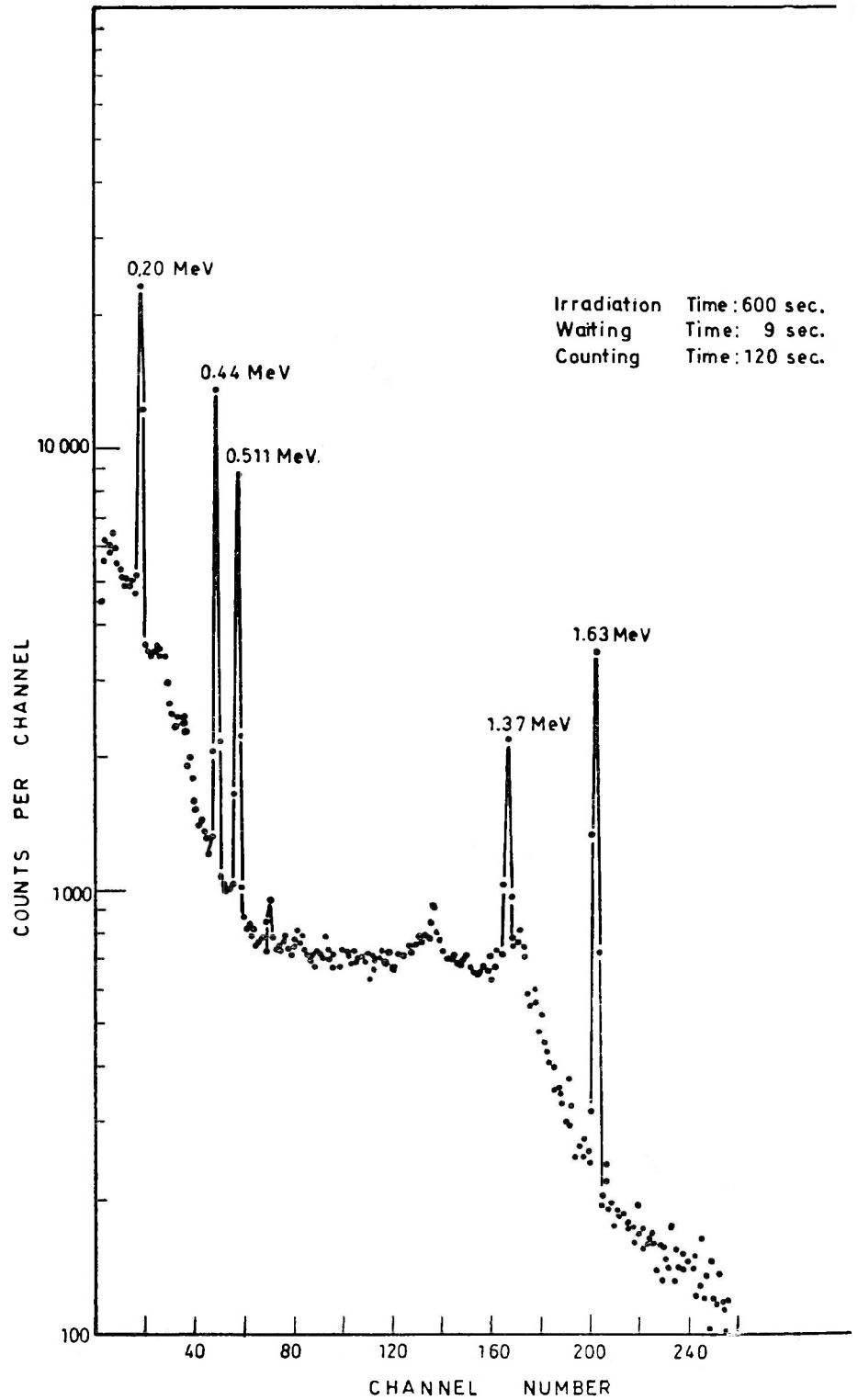

Fig. 1 — Gamma-ray spectrum of NaF

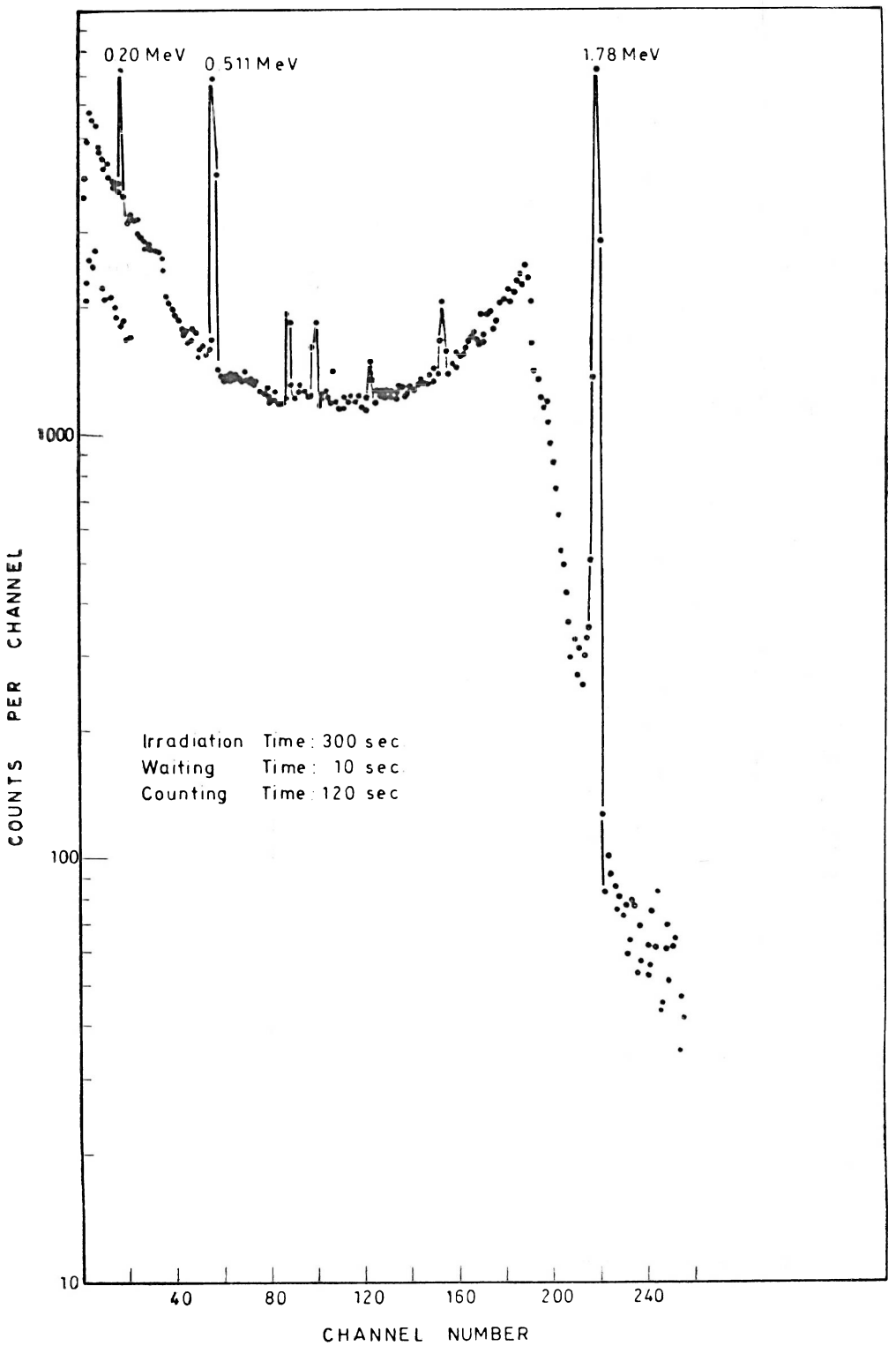

Fig. 2 — Gamma-ray spectrum of apatite

TABLE — 2. The fluorine amount determined in apatite samples by fast neutron activation analysis.

Sample		Fluorine found	
No.	Weight (gr)	Gram	% wt
17908	3.6254	0.4764 ± 0.0101	13.14 ± 0.28
19028	3.0520	0.1203 ± 0.0063	3.94 ± 0.20
CaF_2	4.1649	2.0306 ± 0.0411	48.75 ± 0.98

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