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THE APPLICATION OF ^{3}He-INDUCED NUCLEAR REACTIONS FOR SENSITIVE CHEMICAL ANALYSIS OF SULFUR, CHLORINE, POTASSIUM AND CALCIUM

Diana M. Lee
(M. S. Thesis)

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THE APPLICATION OF ^{3}He-INDUCED NUCLEAR REACTIONS FOR SENSITIVE CHEMICAL ANALYSIS OF SULFUR, CHLORINE, POTASSIUM AND CALCIUM

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THE APPLICATION OF ^{3}He-INDUCED NUCLEAR REACTIONS FOR SENSITIVE CHEMICAL ANALYSIS OF SULFUR, CHLORINE, POTASSIUM AND CALCIUM

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November 1972

ABSTRACT

Three easily-detected radionuclides, ^{38}Cl, ^{34m}Cl, and ^{38}K, produced from ^{3}He reactions on sulfur, chlorine, potassium, and calcium, were used as "signals" for the determination of the latter elements. This method of activation analysis is capable of determining the above elements individually at the microgram level or submicrogram level in some cases. A number of samples were analyzed non-destructively by using differing nuclear reactions to examine the accuracy of the method and the extent of interferences.

Experimentally determined absolute excitation functions have been measured in the ^{3}He energy range from 3 to 22 MeV for the following nuclear reactions: ^{32}S(^{3}He,p)^{34m}Cl, ^{35}Cl(^{3}He,α)^{34m}Cl, ^{39}K(^{3}He,2α)^{34m}Cl, ^{37}Cl(^{3}He,2p)^{38}Cl, ^{37}Cl(^{3}He,2n)^{38}K, ^{39}K(^{3}He,α)^{38}K, and ^{40}Ca(^{3}He,αp)^{38}K.

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INTRODUCTION

Activation analysis is a method of elemental analysis based upon induced nuclear reactions; in many instances it is a very sensitive, non-destructive method for determining a wide variety of elements and isotopes. At present, reactor-produced thermal neutrons are the most commonly used particles for activation analysis; this technique gives mainly (n,γ) nuclear reactions, and will result in small cross sections for many low Z-elements. Thus, neutron activation analysis has limited sensitivity in the case of low-Z elements. Many analytical problems involving trace analysis of low-Z impurities in ultra pure or fissionable materials can be solved non-destructively by using charged-particle activation techniques.

The unique advantages which the ^3He ion offers as an incident particle for activation analysis were first suggested by Markowitz and Mahony[1]. Since then considerable effort has been devoted to developing the practicality of this system for solving various analytical problems[2-7]. Because of the low binding energy of the ^3He nucleus, many ^3He nuclear reactions can be induced with moderate incident energy, giving rise to many easily detectable products with high sensitivity. Another advantage of ^3He reactions is that the Coulomb electrostatic repulsion of high-Z elements prevents interactions with ^3He charged-particles. Therefore, interferences from activated high-Z elements in the matrix can be avoided by the proper selection of the irradiation energy and reaction. However, it should be noted, that some activation can be obtained at particle energies below the classical Coulomb barrier due to the tunneling effects.

Although ^{3}He activation analyses of oxygen, carbon, and fluorine have been investigated extensively in recent years[8-12], there is much less information available about the ^{3}He reactions with sulfur, chlorine, potassium, and calcium. Activation of S, Cl, K, and Ca by thermal neutrons, fast neutrons, protons, deuterons, and alpha particles has been reported[13-19]; most of these analyses were applied to a specific matrix with various degrees of success. The use of ^{3}He ions provides an alternative method for the determinations of the above elements with high sensitivity and a more convenient one in some cases, particularly at low energy.

In order to utilize the charged particle activation technique to its optimum, measurement of activation cross sections for a specific nuclear reaction is essential. Since charged particles lose energy rapidly within the sample, the variation of cross sections as a function of particle energies has to be known. Such information would enable one to make a proper choice of the bombarding energy in order to maximize the yield of the sought activities and at the same time to minimize the interfering activities due to interactions of other elements which may be present in the same sample. This knowledge of excitation functions is also valuable for the productions of radionuclides and for studies of nuclear reaction mechanisms.

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EXPERIMENTAL PROCEDURES

1. Sample Preparation and Assembly

For the measurements of the excitation functions the targets used were prepared by vacuum-depositing CaF_2, KI, $PbCl_2$, and pure sulfur onto tantalum backing-foils. Sulfur targets were protected by depositing a thin film of aluminum over the sulfur to promote heat transfer during the irradiations. The thickness of the targets was determined by weighing; they ranged from 1.5 to 4.5 mg/cm^2. For the subsequent analyses, samples were synthesized as thin targets or as thick ones. Thin targets were prepared either by vacuum evaporation or by settling the finely-ground powder onto a tantalum backing-foil from an ether suspension; the powder was fixed to the foil with a drop of dilute polystyrene in dichloroethylene. The thick targets were prepared by homogenizing a known, small amount of material to be analyzed with pure lead powder (approx. 200 mg/cm^2) and then by pressing the mixture into a 1-inch (2.54 cm) diameter thin disk. Targets were mounted on an aluminum ring and were covered with thin gold or aluminum foils to form one target assembly. This target assembly was constructed to facilitate the handling of a fragile target, to promote heat transfer, to avoid recoil loss, and to obtain a proper, reproducible geometry.

2. Irradiation

Irradiations were performed at the Berkeley 88-inch cyclotron. Aluminum foils were placed in front of the sample assembly to degrade the beam from an initial energy of 30-MeV ^{3}He to an appropriate energy for

each irradiation. The sample assembly was mounted in a conventional water-cooled, target holder. The length of bombardment varied between 8 to 15 minutes according to the product nuclide being studied. The range of beam currents was about 0.1 μA to 0.4 μA of ^{3}He (++). These irradiation conditions produce suitable amounts of the activities for convenient detection. Thin targets were irradiated simultaneously at different energies by stacked-foil technique; thick targets were irradiated, one at a time, with a thin standard in the same stack, or with a thick standard from two successive irradiations. The total charge received from the Faraday cup was measured by a calibrated integrating electrometer.

3. Radioactivity Measurements and Data Acquisition

After irradiation, samples were analyzed by gamma-ray spectrometry, using Ge(Li) detectors, coupled to a 1024-channel analyzer with a magnetic tape recording unit.

During the past several years, lithium-drifted germanium detectors of relatively large active volume have become practical tools for the quantitative measurement of gamma rays. Although these devices have lower efficiencies than the large NaI(Tl) scintillators, the high resolution of Ge(Li) crystals has been found to be of great advantage, particularly in the case of non-destructive activation analysis. Two Ge(Li) detectors, having active volumes of 16 cm^3 and 30 cm^3, manufactured by the nuclear detector group at LBL, were used for this work. The gamma-ray photopeak efficiencies were calibrated using a set of eight absolute gamma-ray standards, obtained from the International Atomic Energy Authority, Vienna.

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The absolute disintegration rates of these IAEA sources were accurate within ± 1%. The gamma-ray energies of the standards cover the energy range from about 60 keV to 1836 keV. The photopeak efficiencies for the gamma rays above this range were extrapolated from a calibrated standard curve.

Data were accumulated for intervals of 4 min. (live-time) for ^{38}K ($T_{1/2}$ = 7.7 min.), 4-10 min. for ^{34m}Cl and ^{38}Cl ($T_{1/2}$ = 32 min. and $T_{1/2}$ = 37 min., respectively). Then the spectral data were recorded on a magnetic tape. Half-lives and gamma-rays used for the measurements are given in Table I. If the high-energy gamma rays were the only ones of interest, a high-rate linear amplifier, coupled to a biased amplifier, which accepted only the high-energy portion of the gamma spectrum, was used to reduce the analyzer dead time; a typical gamma-ray spectrum is shown in Fig. 1. The Ge(Li) detectors were also shielded with lead to assure very low background counting.

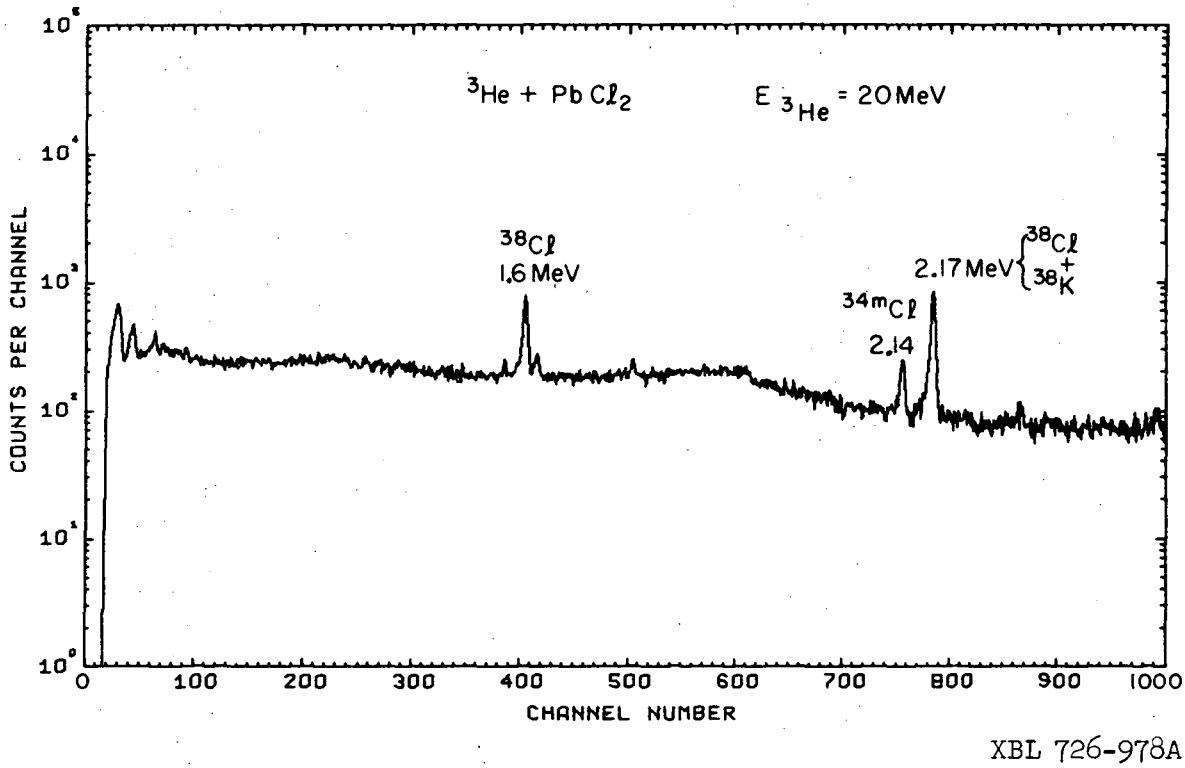

Fig. 1. Gamma-ray spectrum of $PbCl_2$ irradiated by 20-MeV ^{3}He ions; excluding gamma-rays of energies below 1 MeV.

Table I. Nuclear Data, Q Value for the Reactions Investigated in the Present Work.

Nuclear Reaction	$T_{1/2}$ of Product Nuclide (min)	Gamma-ray Energies (1% Intensity)	Gamma-rays Used for Measurement (MeV)	Q Value (MeV)
$^{32}S(^{3}He,p)^{34m}Cl$	32.5	0.145(45%), 0.511(100%), 1.17(12%), 2.12(38%), 3.30(12%)	2.12	+6.08
$^{35}Cl(^{3}He,\alpha)^{34m}Cl$	32.5	"	"	+7.94
$^{39}K(^{3}He,2\alpha)^{34m}Cl$	32.5	"	"	-5.70
$^{37}Cl(^{3}He,2p)^{38}Cl$	37.1	1.60(38%), 2.17(47%)	2.17	-1.61
$^{37}Cl(^{3}He,2n)^{38}K$	7.7	0.511(200%), 2.170(100%)	"	-4.19
$^{39}K(^{3}He,\alpha)^{38}K$	"	"	"	+7.49
$^{40}Ca(^{3}He,\alpha p)^{38}K$	"	"	"	+0.84

CALCULATIONS AND DATA ANALYSIS

1. Thin Target Excitation Functions

The variations of a particular reaction cross section with incident particle energy is called an "excitation function". For a charged-particle bombardment, the mathematical relationship between the disintegration rate of an induced nuclide and the cross section of a thin target is given by

$$D_o = n \sigma I (1-e^{-\lambda t}) \qquad (1)$$

where

σ = reaction cross section in cm^2

D_o = disintegration rate of product at the end of irradiation, disintegration per minute (dpm)

n = number of target atoms per cm^2 of the specific nuclide of interest

I = average beam intensity, in incident ^3He ions per minute

t = length of bombardment, min.

λ = decay constant of induced activity.

The photopeak counting rate at the end of the irradiation, A_o, is given by

$$A_o = (D_o) (ODC) \quad , \qquad (2)$$

where ODC is the Overall Detection Coefficient, it includes detector efficiency, detector geometry, and decay-scheme corrections.

The excitation functions for the total production of the individual product nuclei are most useful for activitation analysis. In this report all excitation functions refer to the total cross section for individual reaction as a function of incident particle energy in the laboratory system.

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The stacked-foil technique was employed in the determination of the excitation functions. The energy of the ^{3}He beam at the appropriate target position in the stack was calculated by applying Steward's computer program[20], which is included as Appendix I for materials used in this work. This code may be employed to calculate ranges of any charged particle in non-gaseous media, pure elements, or compounds. Range energy values for ^{3}He charged particles calculated by utilizing this program agree well with the tabulations of Williamson, et al.[21].

Absolute cross sections were calculated based on the assumption that the reactions in Table I were attributed entirely to a single reaction of a specific target nuclide. The target isotopic abundance, the half-life of product nuclide, its decay mode, gamma-ray energy, and percent gamma-ray decay were taken from the Table of Isotopes[22].

2. Gamma-ray Photo-peak Analysis by Using Computer Program SAMPO:

The resulting radioactive intensities are related to the corresponding gamma-ray photopeak intensities. The primary results of the analysis of gamma-ray spectra are the energies and intensities of the photopeaks and the estimated precision. The energies are related to channel locations through energy calibration, and the intensity of peak areas through efficiency calibration. The precision is determined by the statistics of the data and by the uncertainties in the calibration procedures.

The FORTRAN IV code SAMPO, was developed by Routti and Prussin[23] for the CDC 6600 computer. This program provides a least-squares fit to the original data points with a line shape and a polynominal approximation

for the continuum. It attempts to fit a gamma-ray photopeak by a Gaussian function with an exponential "tail" on both sides. This code can also be used to resolve multiplets. The parameters defining the line shape were obtained by interpolating linearly the results of the shape analysis of the calibration peaks, or by a standard known peak from the same detector system. In this work, fitting close-energy peaks in a high energy region (^{34m}Cl = 2.14 MeV, ^{38}Cl = 2.17 MeV) was found to be very convenient by using peak parameters from sources containing only one isolated peak of the same energy. A single gamma-ray photopeak of ^{34m}Cl produced from ^{3}He bombarding Cl target at about 7-8 MeV was taken to obtain the peak shape parameters for the high energy region.

This program SAMPO has been designed primarily for an off-line analysis with a large amount of internal decision-making, but it can also be run in an interactive on-line mode employing cathode-ray-tube (CRT) display, teletype, and light-pen input. For this work, a combination of both automatic off-line and interactive on-line analysis was employed to promote a rapid data reduction procedure of high accuracy and with minimum computer time.

3. Decay-Curve Analysis

A least-square computer code, CLSQ[24], was used to resolve a composite of exponential decay curves into its components. This code can yield iterative solutions for half-lives and A_o values. For most of the present work, multicomponent decay-curve analysis was not necessary because the high energy gamma-rays from well-resolved single components were used instead of an annihilation radiation. In the case of ^{38}Cl + ^{38}K produced in the same sample, both emitting 2.17 MeV gamma ray with different

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half-lives, decay-curve analysis was used to obtain a more accurate A_o value for each of the components. This computer code is also useful for obtaining a more accurate A_o value for single-component decay curves; it also calculated the standard deviation with appropriate statistical weight.

4. Sample Analysis: Absolute Analysis and Comparison Analysis

Both absolute and comparison analyses were used in this experiment. In the absolute analysis, Eqs. (1) and (2) are reexpressed as

$$n = \frac{A_o}{(ODC)\,\sigma\, I\, (1 - e^{-0.693\, t/T_{1/2}})} \qquad (3)$$

and the mass of target nuclei is related to n and A, the mass number, by the formula

$$\text{mass}(g/cm^2) = \frac{nA}{6.025 \times 10^{23}} \qquad (4)$$

All parameters in Eq. (2), except n, were known or were determined during the experiment. In general, σ is a function of the energy of the charged particles; however, the energy of the beam is degraded only slightly in a thin target, so that σ in Eq. (2) can be considered constant. Thus, in practice, sample thickness is restricted such that the variation in cross section within the sample will permit measurements to a chosen limit of accuracy. Absolute analysis was also performed for some very thick samples which completely stop the incident beam. In this case, an average cross section, $\bar{\sigma}$, replaced σ in Eq. (2). Ricci and Hahn (2,3) defined this "thick target average cross section", $\bar{\sigma}$, by

$$\bar{\sigma} = \frac{1}{R} \int_{o}^{R} \sigma_t \, dt \tag{5}$$

where R is the total range of the incident ^{3}He ion in the target material and σ_t is the cross section as a function of target thickness. They have shown that $\bar{\sigma}$ is independent of the target matrix and supported the calculations with experimental evidence.

A comparison standard method was applied to samples of various thickness. For charged-particle activation analysis, the general form of mathematical expression is

$$\frac{A_x^o}{A_s^o} = \frac{ODC_x N_x \sigma_x I_x (1 - e^{-\lambda t_x})}{ODC_s N_s \sigma_s I_s (1 - e^{-\lambda t_s})} \tag{6}$$

where x signified unknown and s stands for comparison standard. The terms are as defined in Eqs. (1) and (2). When a thin sample and a thin standard procedure was used in successive irradiations, Eq. (6) reduced to

$$\frac{A_x^o}{A_s^o} = \frac{N_x I_x}{N_s I_s} \tag{7}$$

for equal irradiation time, irradiation energy and counting geometry; the only correction necessary was made for the differences in I_x and I_s.

For the intermediate thickness samples, a known composition standard was prepared to match the degrading power of the sample. Very often such standards were not available; in those cases a correction factor obtained from the excitation function for the reaction was applied to account for the difference in the average cross sections.

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For homogeneous thick targets, the average cross sections for both standard and sample were approximately equal, but the number of target nuclei in the sample and standard depends upon the range of the beam in the target matrix, in this instance Eq. (6) becomes

$$\frac{A_x^o}{A_s^o} = \frac{ODC_x N_x R_x I_x (1 - e^{-\lambda t_x})}{ODC_s N_s R_s I_s (1 - e^{-\lambda t_s})} \qquad (8)$$

where R_x and R_s are the ^3He ion ranges in unknown and standard matrices. Eq. (8) can also be reduced to

$$\frac{A_x^o}{A_s^o} = \frac{N_x R_x I_x}{N_s R_s I_s} \qquad (9)$$

for equal irradiation time, irradiation energy, counting geometry.

5. Limits for Quantitative Determination

The detailed mathematical and statistical treatment of the limits for qualitative detection and quantitative determination in their application to radiochemistry has been discussed by Currie[25]. Only the limit for quantitative determination will be considered in this work. For quantitative analysis a general expression, relating determination limit in mass to the determination limit in activity by means of Eq. (10),

$$\text{determination limit (g)} = \frac{\text{determination limit (activity)}}{\text{total counts}} \qquad (10)$$

is applicable. A "working expression", derived by Currie for the determination limit (activity) with 10% precision, is given by,

$$L_q = 50 \left\{ 1 + \left[1 + \frac{\mu_B}{12.5} \right]^{1/2} \right\} \qquad (11)$$

where L_q is the determination limit (count) and μ_B is background counts per counting interval. For very low background counting, L_q is approximately 100 counts. The total counts in Eq. (10) can be estimated by the counting interval and by the initial activity by means of Eq. (12),

$$\text{total counts} = A_o \, (e^{-\lambda t}/\lambda) \, (1 - e^{-\lambda \Delta t}) \qquad (12)$$

where A_o = disintegration rate at the end of irradiation

λ = decay constant of the nuclei

t = delay or cooling time

Δt = counting interval .

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RESULTS AND DISCUSSION

1. Excitation Functions

A summary of experimental cross-section data obtained in this work is presented in Table II and the excitation functions for the several elements are shown in Figs. 2 through 5. Each of the cross sections shown in Table II represents an average value of 2-3 replicates; the precision (average deviation) was about 5%. Additional uncertainties include: (1) the determination of total beam current, (2) the half-lives of nuclides, (3) the determination of disintegration rates including decay-curve analysis, counter calibration, counter instability, (4) the uniformity of thickness of targets, and (5) the possibility of recoil gas losses during irradiation. Comprising the above considerations, the estimated absolute errors in the cross sections are 10% for S data, 10-15% for Cl, 20% for K, and 10% for Ca. The uncertainty in the initial beam energy of 30-MeV ^{3}He was estimated to be $\pm$ 0.15 MeV. As the beam traverses the degraders and the target materials, the spread of beam energies and the range energy calculation may contribute to about $\pm$ 1 MeV of uncertainty at beam energy of 10 MeV.

Not all the excitation functions investigated in this work have reached their maximum cross section at 20-MeV beam energy. For the reactions (^{3}He,2p), (^{3}He,2α), and (^{3}He,αp), maximum cross sections would be expected to occur at higher energies.

2. Interferences

There are two types of interferences in non-destructive activation analysis in conjunction with gamma-ray spectrometry. The first type of

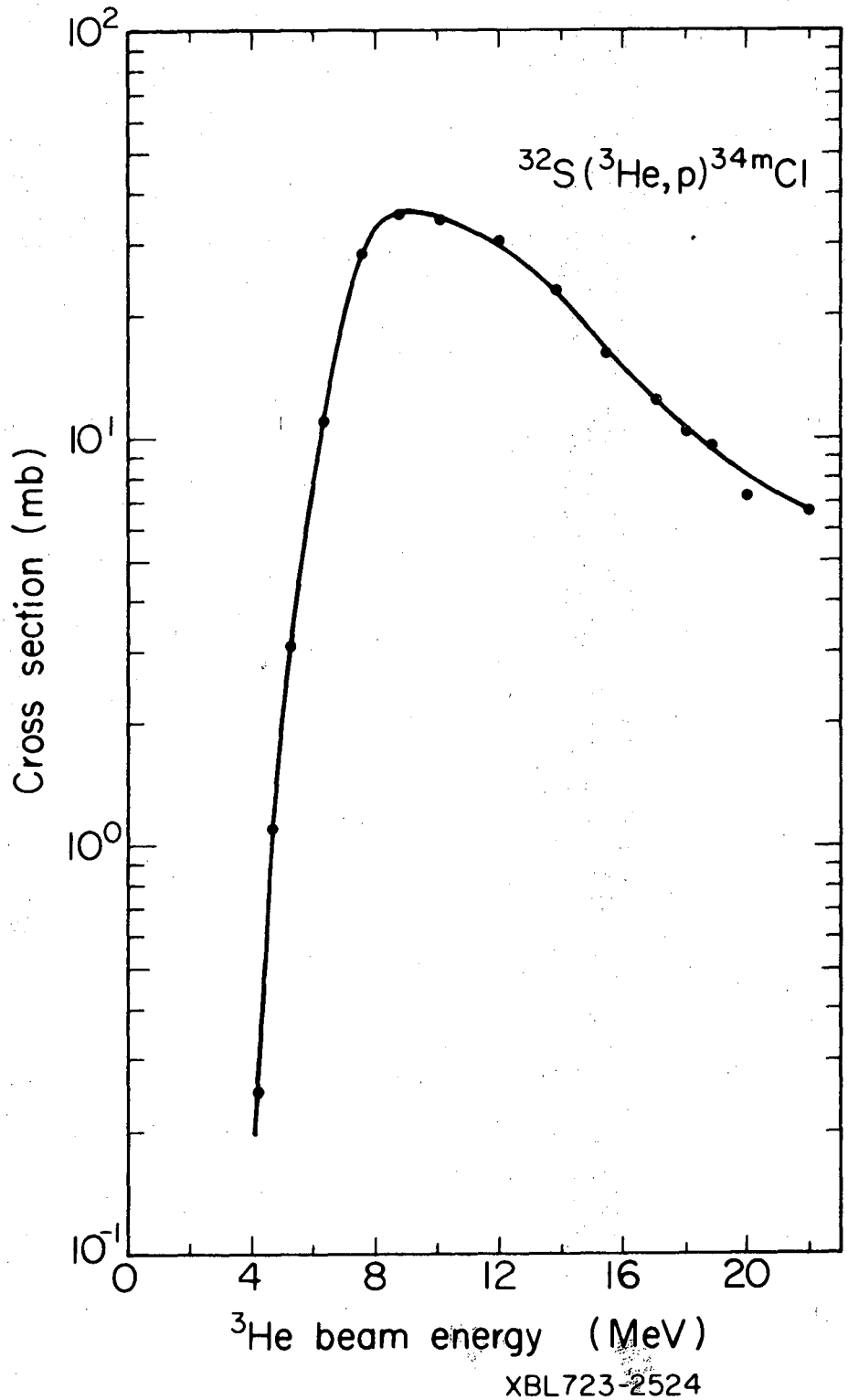

Fig. 2. Excitation function for the reaction $^{32}S(^3He,p)^{34m}Cl$.

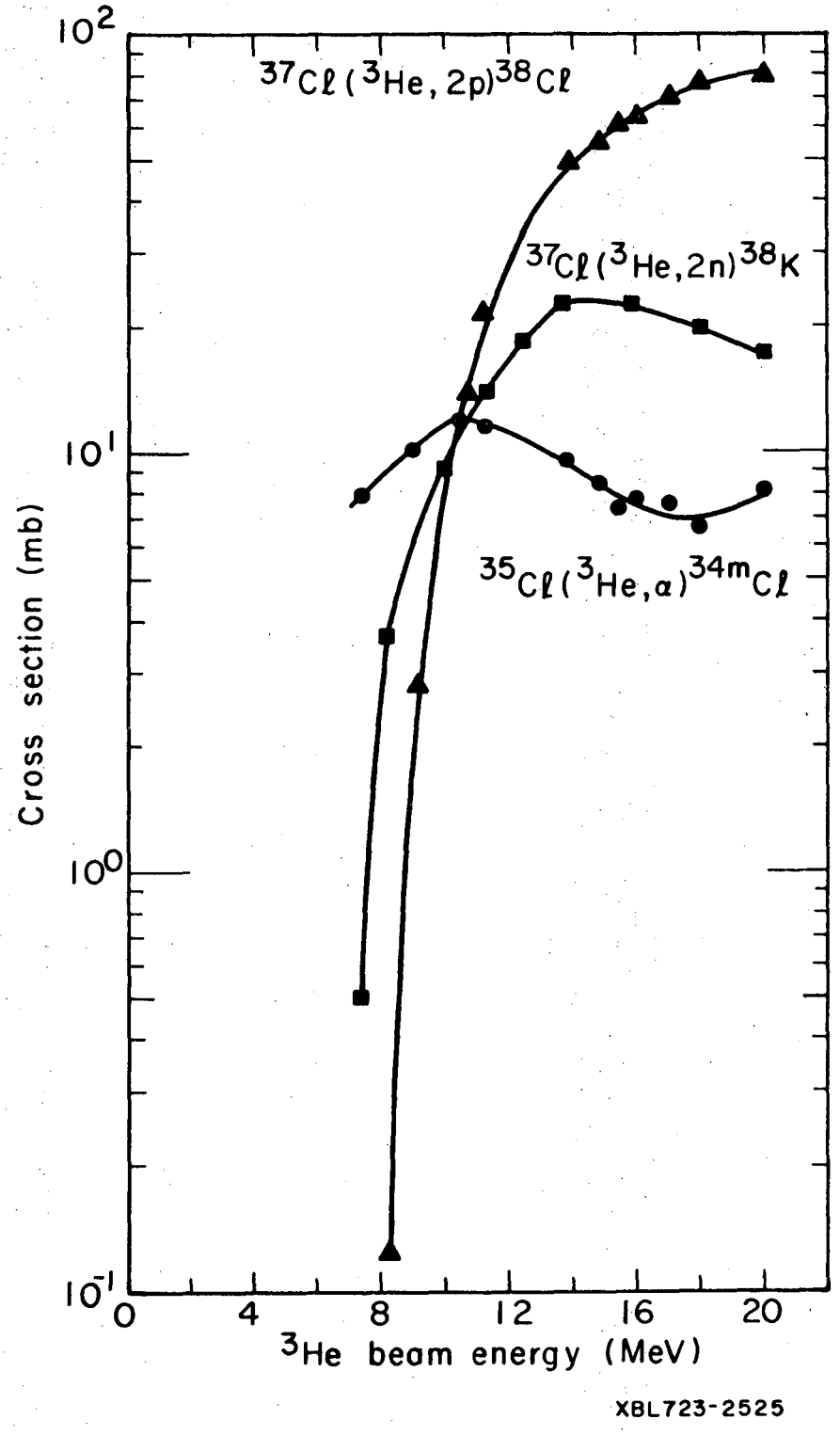

Fig. 3. Excitation functions for reactions ^{3}He + Cl. ▲ $^{37}Cl(^3He,2p)^{38}Cl$ $^{37}Cl(^3He,2n)^{38}K$ $^{35}Cl(^3He,\alpha)^{34m}Cl$.

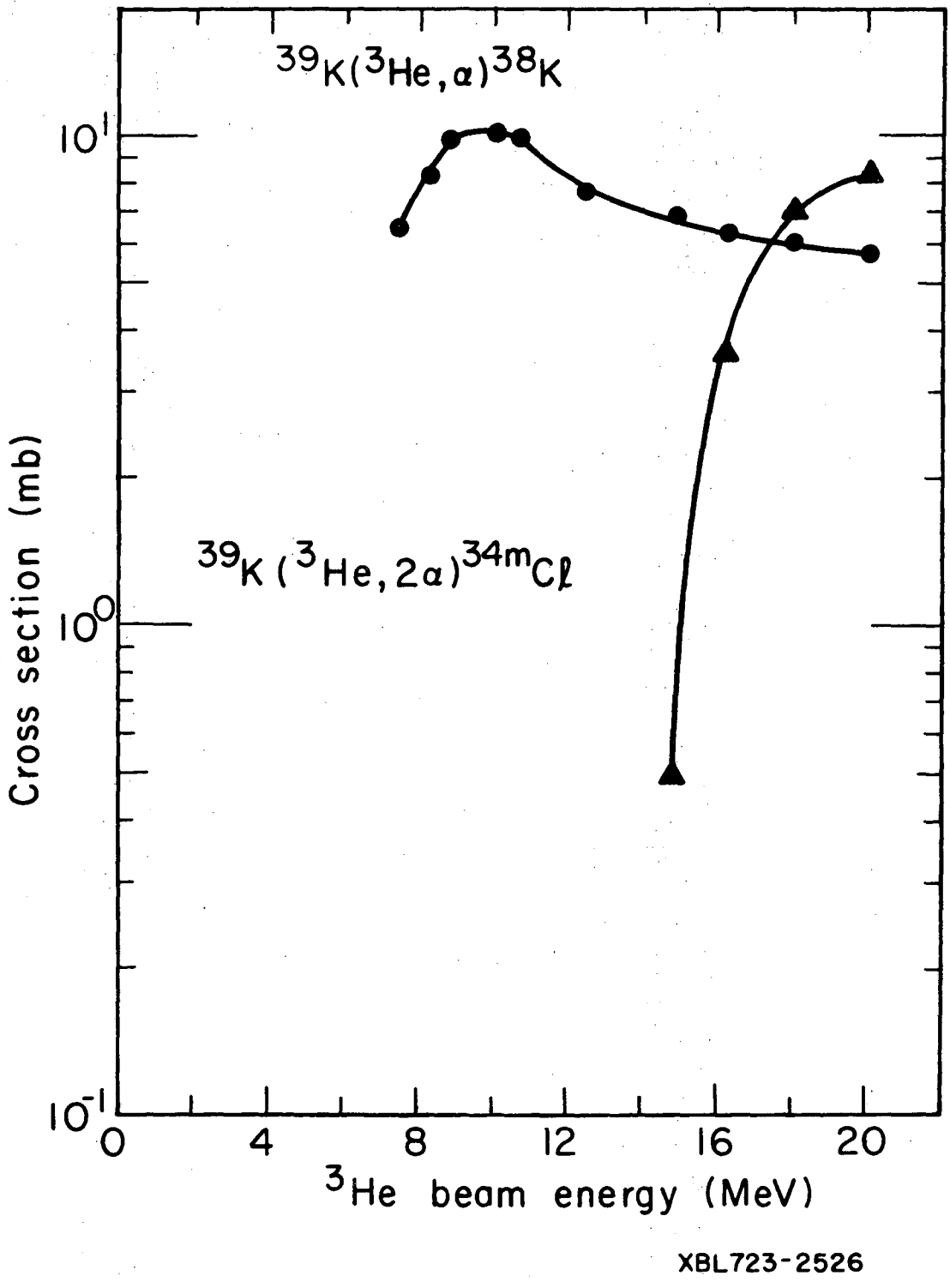

Fig. 4. Excitation functions for reactions ^{3}He + K. ● $^{39}K(^{3}He,\alpha)^{38}K$. ▲ $^{39}K(^{3}He,2\alpha)^{34m}Cl$.

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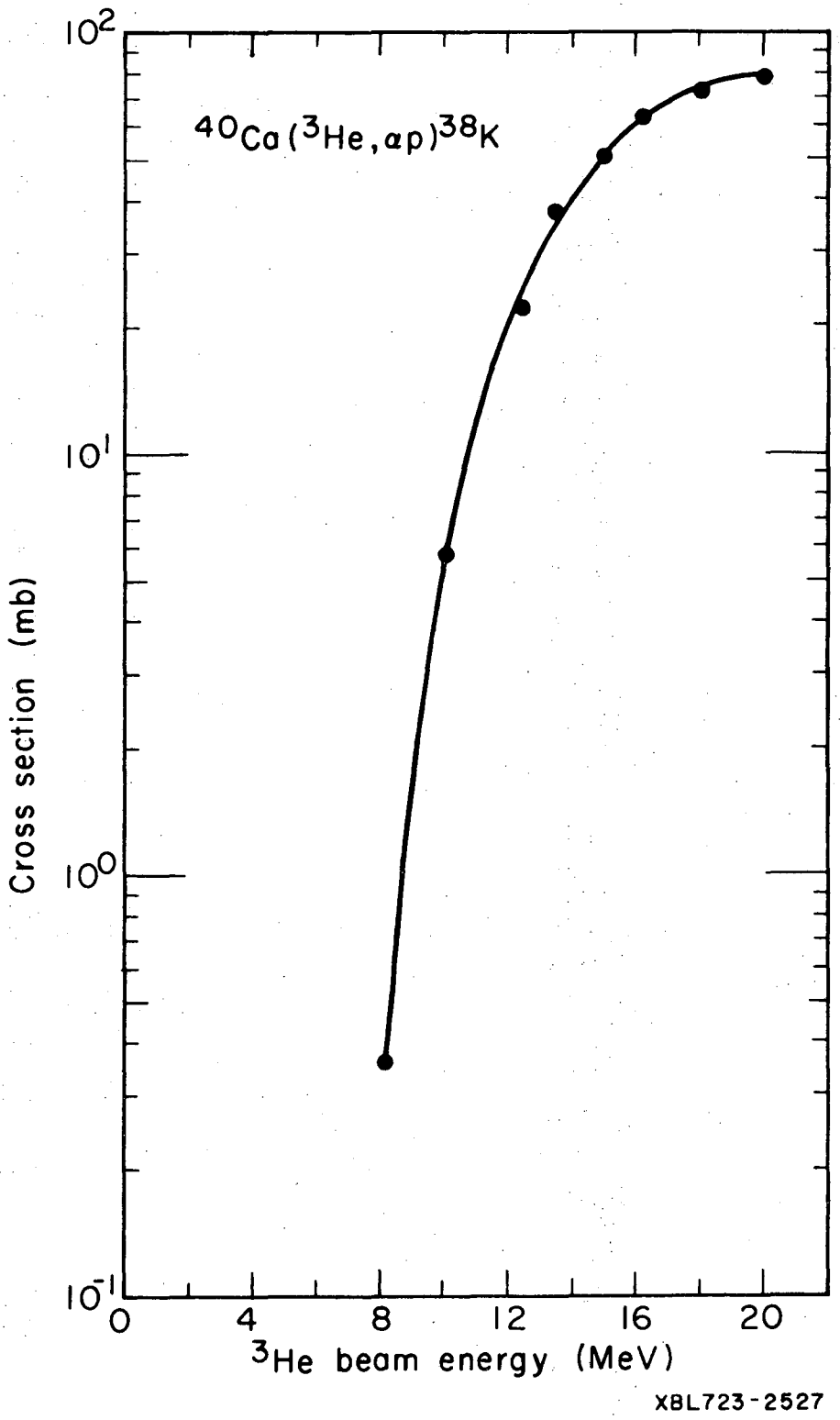

Fig. 5. Excitation function for the reaction $^{40}Ca(^3He,\alpha p)^{38}K$.

Table II. Cross Sections (in mb) for ^{3}He Induced Nuclear Reactions on S, Cl, K, and Ca.

Average Beam Energy (MeV)	Energy Interval (MeV)	^{32}S (^{3}He,p) ^{34m}Cl	^{35}Cl (^{3}He,α) ^{34m}Cl	^{37}Cl (^{3}He,2p) ^{38}Cl	^{37}Cl (^{3}He,2n) ^{38}K	^{39}K (^{3}He,α) ^{38}K	^{39}K (^{3}He,2α) ^{34m}Cl	^{40}Ca (^{3}He,αp) ^{38}K
4.2	4.6-3.8	0.25						
4.6	5.0-4.3	1.1						
5.2	5.6-4.9	3.1						
6.3	6.6-6.1	11.1						
7.5	7.8-7.3	28.4	7.9	0	0.5	6.4		
8.2	8.4-8.1			0.1	3.8	8.2		0.36
8.7	9.0-8.4	35.2				9.8	0	
8.9	9.2-8.6		10.3	2.8				
10.1	10.3-9.4	34.2			9.1	10.2		5.9
10.6	10.9-10.4		12.1	13.4	9.3	9.9		
11.2	11.5-11.0		11.6	21.8	12.9			
11.9	12.1-11.6	31.0						
12.4	12.6-12.3				18.3	7.7	0	22.4
13.8	14.0-13.6	23.1	9.5	48.0	22.8			39.1

(continued)

Table II (cont.)

Average Beam Energy (MeV0	Energy Interval (MeV)	^{32}S (^{3}He,p) ^{34m}Cl	^{35}Cl (^{3}He,α) ^{34m}Cl	^{37}Cl (^{3}He,2p) ^{38}Cl	^{37}Cl (^{3}He,2n) ^{38}K	^{39}K (^{3}He,α) ^{38}K	^{39}K (^{3}He,2α) ^{34m}Cl	^{40}Ca (^{3}He,αp) ^{38}K
14.8	15.0-14.7		8.5	53.1	23.0	6.9	0.5	50.5
15.4	15.6-15.3	16.2	7.4	60.5				
15.9	16.0-15.7		7.8	62.2	22.6			
16.2	16.4-16.0					6.3	3.6	62.6
17.1	17.3-17.0	12.4	7.7	70.0				
18.0	18.1-17.8	10.3	6.7	75.0	20.1	6.1	7.1	72.1
18.8	19.0-18.6	9.7						
20.0	20.2-19.9	7.2	8.1	77.7	17.2	5.8	8.2	76.6
22.0	22.1-21.8	6.7						

interference is due to the "closely-spaced" gamma-ray, or to gamma-rays having the same or similar energies; the second kind is caused by the production of the same nuclide from more than one element. Interference of the first type may be minimized by using special counting techniques such as coincidnece or anti-coincidence counting, or by using a high resolution Ge(Li) gamma-ray detector system. One can also resort to radiochemical separation of desired radionuclides, emitted from the same nuclide; the choice of selecting a particular gamma-ray depends on two important factors; (a) the relative intensity and (b) the presence of other gamma-rays. In view of these interference possibilities, gamma-rays of high-energy were chosen for the measurements of ^{38}Cl, ^{34m}Cl, and ^{38}K in the present work.

Relative activities of the same reaction product-nuclide formed from more than one element in the activations of S, Cl, K, and Ca are shown in Table III and IV. Chlorine can be determined unambiguously by detection of ^{38}Cl from the $^{37}Cl(^{3}He,2p)^{38}Cl$ reaction, because no other element will produce ^{38}Cl under our conditions. However, for the determination of Ca, K, and S, the interfering activities, if present, can not be completely eliminated. It is possible to minimize these interferences by the adjustment of bombarding energies. Table III shows the yield of the ^{38}K activity from calcium which is nearly 9 times as much as the combined yield from potassium and chlorine for equal weight of natural elements on a thin sample and at an irradiation energy of 20 MeV. Nevertheless, at an energy of 8 MeV, K can be determined with small interferenc from Cl and Ca. The error in the determination of calcium due to potas

0 0 0 0 3 8 0 0 1 2 9

and chlorine will be approximately 10% for samples containing equal amounts of calcium, potassium and chlorine. Yet, the extent of interference can be assessed by knowing the ratio of production from the interfering element. For example, to determine Ca in the presence of K and Cl at 20-MeV ^{3}He irradiation energy, the ^{38}K activity produced from K and Cl can be estimated by the activity ^{34m}Cl and ^{38}Cl. For the determination of S, the best result was obtained at the energy of 10 MeV, at which no interference from K occurred and at which the amount of interference from Cl could be estimated by the activity of ^{38}Cl (both ^{34m}Cl and ^{38}Cl photopeaks could be observed simultaneously).

3. Sensitivity of the Method

The sensitivity of the method may be predicted by considering the systematic parameters which govern the production of activities from sought elements in an easily obtainable condition. The parameters are the incident beam energy, the beam current, the counting efficiency, the counting interval, and the duration of irradiation. The count rate of the product activities per gram of the sought element at the end of the irradiation (A_o) can be calculated by using Eq. (3); σs of Eq. (3) are obtained from Table II for various beam energies. Then, the total number of counts accumulated during a counting interval (Δt) is calculated using Eq. (12). Applying Eq. (10) for the interference-free determinations of S, Cl, K, and Ca by activation with ^{3}He at the energy of the maximum cross section, using 1 μA current for one half-life irradiation, and by employing a

photopeak counting efficiency of only about 0.5%, we estimate the determination limits in a 1-gram sample to be 0.025 μg for S, 0.05 μg for Cl, 0.2 μg for K, and 0.025 μg for Ca.

4. Results of Sample Analyses

Samples containing compounds with known amount of S, Cl, K, and Ca were analyzed non-destructively. The A_o values (activity at the end of bombardment) were determined by following the gamma-ray photopeaks for 5 to 6 half-lives; the intensity of the photopeaks was analyzed by computer code SAMPO and the A_o values were obtained by decay-curve analysis using CLSQ code. For the comparison method, the amount of elements present was calculated by comparing the ratio of the A_o values of the samples to the appropriate standards. The standards used for the analyses of S, Cl, K, and Ca in this work were pure S, $PbCl_2$, KI, and CaF_2 respectively, having thickness similar to the samples. The absolute method also has been used for several thin samples. Concentrations of several samples were verified by wet chemical analysis. The results of the sample analyses are shown in Table V; the average deviation from the known concentrations of all samples is about 6%. Many commonly-present elements, such as Na, Al, Pb, Ag, Si, and F, did not interfere in the analyses of S, Cl, K, and Ca. Results also show that in the determination of Cl in many samples, ^{38}K, ^{38}Cl, and ^{34m}Cl could be used with approximately the same accuracy.

Table V. Results of ^{3}He Activation Analyses.

Sample	Element Sought	^{3}He Beam (MeV)	Nuclide Used	Element, mg/cm^2 Present	Found[a]	Dev. from Known (%)
S-1	S	10	^{34m}Cl	0.21	0.20	-4.7
S-2	S	10	^{34m}Cl	0.54	0.52	-3.7
S-3	S	10	^{34m}Cl	1.09	1.14	+4.5
S-4+Pb	S	15	^{34m}Cl	0.56	0.61	+8.9
S-5+Pb	S	15	^{34m}Cl	1.12	1.20	+7.0
Saran-1	Cl	15	^{38}Cl	1.20	1.13	-5.8
Saran-2	Cl	15	^{38}K	0.99	0.95	-4.0
AgCl-1	Cl	10	^{34m}Cl	0.75	0.80	+6.6
AgCl-2	Cl	15	^{38}K	0.75	0.78	+4.0
NaCl-1	Cl	15	^{38}Cl	0.21	0.20	-4.7
NaCl-2	Cl	15	^{38}K	1.08	1.06	-1.8
$PbCl_2$+Pb	Cl	15	^{38}Cl	0.10	0.09	-10
$PbCl_2$+Pb	Cl	15	^{38}K	0.10	0.09	-10
KCl-1	Cl	15	^{38}Cl	1.35	1.31	-2.9
KCl-1	K	8	^{38}K	1.48	1.41	-4.7
Mica-1	K	10	^{38}K	0.09	0.08	-11
Mica-2	K	10	^{38}K	0.07	0.06	-14
Mica-3	K	20	^{38}K	0.18	0.16	-11
CaF_2-1	Ca	20	^{38}K	0.54	0.55	+1.8
CaF_2-2	Ca	20	^{38}K	0.26	0.27	+3.8
CaF_2-3	Ca	20	^{38}K	0.13	0.14	+7.7

[a]Average of 2 measurements. Av. Dev. 6%

CONCLUSION

The investigation of excitation functions of reactions producing ^{38}Cl, ^{38}K, and ^{34m}Cl from Cl, S, Ca, and K has permitted us to estimate interferences as a function of the ^{3}He energy. The interference-free sensitivities in 1-gram samples were estimated to be 0.025 μg for S; 0.05 μg for Cl; 0.2 μg for K; and 0.025 μg for Ca (for a beam current of 1 μA, and irradiation time of 1 half-life).

A comparison of Cl analysis by use of three different reaction products has been used to demonstrate the flexibility of ^{3}He-induced reactions for activation analysis. The application of ^{3}He activation analysis also has been demonstrated with various samples to an average accuracy of 6% for the elements S, Cl, K, and Ca.

The method can usually be successfully applied for determining above elements non-destructively in samples such as Na, Al, Pb, Ag, Si, and F.

0 0 0 0 3 8 0 6 1 3 1

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APPENDIX I

Range-Energy Table for ^{3}He Ions

The ranges of 0-30 MeV ^{3}He ions in nongaseous targets are given for elements and compounds relevant to the present work. The range data were calculated using the computer program of P. G. Steward[20]. This computer program uses a combination of experimental data, the nuclear and electronic stopping-power theory developed by Lindhard[26] et al., and Bethe's theory[27] for ions at high energy.

RANGE OF HE-3 IONS INCIDENT ON NON-GASEOUS TARGETS

ENERGY MeV	RANGE (mg/cm^2) NaCl	KCl	AgCl	$PbCl_2$	Pb
0.5	0.64	0.69	1.20	1.57	2.78
1.0	1.09	1.18	2.02	2.59	4.34
1.5	1.61	1.74	2.91	3.68	5.94
2.0	2.20	2.36	3.91	4.88	7.65
2.5	2.90	3.10	5.07	6.25	9.54
3.0	3.61	3.86	6.26	7.66	11.47
3.5	4.54	4.82	7.74	9.40	13.79
4.0	5.46	5.79	9.23	11.14	16.10
4.5	6.38	6.76	10.71	12.87	18.41
5.0	7.52	7.95	12.50	14.96	21.12
5.5	8.65	9.14	14.28	17.04	23.82
6.0	9.80	10.33	16.07	19.12	26.53
6.5	11.23	11.82	18.26	21.66	29.77
7.0	12.66	13.31	20.44	24.20	33.01
7.5	14.09	14.80	22.62	26.73	36.25
8.0	15.52	16.29	24.81	29.26	39.49
8.5	16.95	17.78	26.99	31.80	42.73
9.0	18.38	19.26	29.17	34.33	45.97
9.5	20.15	21.10	31.82	37.38	49.81
10.0	21.92	22.94	34.45	40.42	53.66
10.5	23.69	24.77	37.09	43.46	57.51
11.0	25.46	26.61	39.73	46.51	61.35
11.5	27.23	28.45	42.37	49.55	65.20
12.0	29.00	30.28	45.01	52.60	69.05
13.0	33.21	34.63	51.16	59.66	77.89
14.0	37.41	38.97	57.30	66.72	86.74
15.0	41.61	43.32	63.45	73.79	95.59
16.0	46.81	48.67	70.89	82.29	106.14
17.0	51.99	54.02	78.33	90.79	116.69
18.0	57.19	59.37	85.77	99.29	127.23
19.0	62.38	64.72	93.20	107.79	137.78
20.0	67.57	70.07	100.64	116.30	148.33
21.0	72.76	75.42	108.08	124.80	158.88
22.0	79.47	82.30	117.44	135.43	171.91
23.0	86.17	89.18	126.80	146.06	184.95
24.0	92.87	96.05	136.16	156.70	197.99
25.0	99.56	102.93	145.52	167.33	211.02
26.0	106.27	109.81	154.87	177.97	224.06
27.0	112.97	116.69	164.23	188.60	237.10
28.0	119.67	123.57	173.59	199.23	250.14
29.0	126.37	130.44	182.95	209.86	267.17
30.0	133.07	137.32	192.31	220.50	276.21

RANGE OF HE-3 ION INCIDENT ON NON-GASEOUS TARGETS

ENERGY MEV	RANGE (mg/cm^2) S	KI	CaF_2	Mica[1]	SARAN[2]
0.5	0.63	1.37	0.63	0.55	0.63
1.0	1.09	2.29	1.08	.94	1.09
1.5	1.60	3.28	1.59	1.39	1.60
2.0	2.18	4.40	2.17	1.91	2.19
2.5	2.87	5.68	2.85	2.52	2.88
3.0	3.58	6.99	3.56	3.16	3.59
3.5	4.48	8.62	4.46	3.98	4.51
4.0	5.39	10.25	5.36	4.79	5.42
4.5	6.29	11.88	6.26	5.61	6.33
5.0	7.41	13.84	7.37	6.62	7.47
5.5	8.53	15.79	8.49	7.63	8.59
6.0	9.65	17.75	9.60	8.65	9.72
6.5	11.06	20.14	10.99	9.93	11.14
7.0	12.46	22.52	12.39	11.21	12.56
7.5	13.86	24.91	13.79	12.49	13.98
8.0	15.27	27.29	15.19	13.77	15.40
8.5	16.67	29.67	16.59	15.05	16.81
9.0	18.07	32.06	17.98	16.33	18.24
9.5	19.81	34.93	19.71	17.92	19.99
10.0	21.54	37.80	21.44	19.52	21.75
10.5	23.28	40.67	23.17	21.11	23.50
11.0	25.01	43.54	24.90	22.70	25.26
11.5	26.75	46.41	26.63	24.30	27.01
12.0	28.49	49.28	28.36	25.89	28.77
13.0	32.60	55.95	32.47	29.69	32.94
14.0	36.71	62.62	36.57	33.50	37.11
15.0	40.83	69.30	40.67	37.30	41.28
16.0	45.90	77.35	45.74	42.02	46.43
17.0	50.98	85.39	50.81	46.74	51.58
18.0	56.06	93.44	55.88	51.47	56.73
19.0	61.14	101.49	60.95	56.19	61.88
20.0	66.21	109.54	66.02	60.91	67.03
21.0	71.29	117.59	71.09	65.63	72.18
22.0	77.84	127.68	77.63	71.77	78.83
23.0	84.38	137.78	84.17	77.90	85.48
24.0	90.92	147.87	90.71	84.04	92.12
25.0	97.47	157.97	97.26	90.17	98.77
26.0	104.01	168.06	103.80	96.31	105.42
27.0	110.55	178.16	110.34	102.44	112.07
28.0	117.10	188.25	116.88	108.58	118.72
29.0	123.64	198.34	123.42	114.71	125.36
30.0	130.18	208.44	129.97	120.85	132.01

[1]Mica: $K_2O \cdot 3Al_2O_3 \cdot 6SiO_2 \cdot 2H_2O$

[2]SARAN: $[CH_2CCl_2]_\eta$

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