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Diana M. Lee, Cynthia V. Stauffacher, and Samuel S. Markowitz

January 1970

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DETERMINATION OF OXYGEN IN COPPER BY ³He ACTIVATION ANALYSIS Diana M. Lee, Cynthia V. Stauffacher, and Samuel S. Markowitz

Department of Chemistry and, Lawrence Radiation Laboratory, University of California, Berkeley, California 94720

January 1970

BRIEF

A radioactivation analysis using low-energy ³He as incident particle and ¹⁸F as "signal" for oxygen present in copper at concentrations in the ppb range or higher is presented. The method is nondestructive, extremely sensitive, and rapid. Only fractions of a milligram of sample are required.

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ABSTRACT

A radioactivation method using ³He as the bombarding particle has been developed to determine trace concentrations of oxygen in copper. The limits of detection extend to about 0.35 parts per billion with a sensitivity of approximately 29 dpm/ppb of radioactive 110-min ¹⁸F which is used as the "signal" for The ¹⁸F is produced by the nuclear reaction of ¹⁶O with low-energy (4 to 10 MeV) 3 He ions. The 0.51-MeV annihilation radiation from the 18 F is detected by a NaI scintillation spectrometer, or by a Ge gamma-ray spectrometer. At ³He energies below 6.3 MeV, no interfering radioactivities from reactions with the Cu matrix are present so the analysis is nondestructive. If necessary, destructive analysis may be carried out and a rapid, clean procedure for radiochemical separation of ¹⁸F as PbF₂ is presented. The method is calibrated either absolutely through measurement of beam intensity, detection coefficient, cross section, and bombardment time, or through a comparative method based upon standards of known oxygen content prepared by anodic oxidation of tantalum foils. The oxygen content of 0.1-mil Cu foil was measured to be 550 ± 30 ppm $(0.055 \pm 0.003\%)$. The relative standard deviation was 5%. Only fractions of a milligram of sample are required. The possibility of continuous analysis using low-energy "desk-top" cyclotrons designed specifically for 3He ions is suggested.

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INTRODUCTION

The properties of copper (and other metals) depend strongly on the content of trace impurities, especially oxygen. Electrical conductivity, for example, may be strongly affected by oxygen even at trace concentrations. The direct determination of trace amounts of oxygen in high-purity metals is a difficult analytical problem. Various methods for oxygen analysis in metals have been tried; among these are vacuum-fusion, carrier-gas fusion, and nuclear activation analysis.

Activation analysis offers a sensitive and nondestructive means of elemental analysis in general. Several nuclear reactions have been applied for oxygen analysis such as (n,p), (t,n), $(^3\text{He},p)$, and $(^4\text{He},pn)$ $(\underline{1-6})$. Among these nuclear reactions, ^3He as the incident particle has been shown to be a sensitive probe for oxygen analysis. ^3He activation analysis in general has been described by Markowitz and Mahony $(\underline{5})$. Other workers have studied different aspects and problems inherent in this technique $(\underline{7-9})$.

By measuring ¹⁸F produced from the reaction

$$^{16}O(^{3}\text{He,p})^{18}\text{F} + ^{16}O(^{3}\text{He,n})^{18}\text{Ne} \longrightarrow ^{18}\text{F}$$
,

several workers ($\underline{10-13}$) have reported analytical methods and results from analyses of microamounts of oxygen in Al, Zr, La, Am, Pu, Si, Au and Tb metals. In addition, determination of the isotopic ratios 18 O/ 16 O by means of 3 He activation in several matrices has been demonstrated ($\underline{14}$). The 18 O was detected through its 1.63-MeV gamma ray produced by the 18 O(3 He,p) 20 F reaction; 16 O was detected through the reaction described in this paper.

Trace oxygen analysis in copper has not yet been obtained by ^3He activation analysis or the other nuclear analyses, due to large interferences from activated copper. Theoretically, nondestructive analysis for oxygen can be achieved by lowering the ^3He bombarding energy below the Coulomb barrier of copper; the Coulomb barrier of copper (Z = 29) for ^3He (Z = 2) is 10 MeV ($r_o = 1.5 \times 10^{-13}$ cm). Byrant, Cochran and Knight (15), however, reported small but appreciable cross sections for some reactions of copper with ^3He at energies below 10 MeV. The excitation function for production of ^{18}F from ^{16}O , Figure 1, (5) shows that the maximum cross section is about 400 mb at ^3He energy of 7.5 MeV. If the incident energy of ^3He is reduced below 6 MeV, production of ^{18}F from ^{16}O will decrease measurably. Using this energy consideration and variation of cross section, we made a study to locate the optimum bombarding energy for maximum sensitivity, freedom from Cu-induced interferences, and accuracy.

A 7.5-MeV ³He ion has a range of 18 mg/cm² in copper. Thus, the depth of penetration is less than 0.001 inch. If it is desired to measure oxygen content over depths greater than 0.001 inch, the ³He bombarding energy should be raised to about 10 MeV, or higher. At higher energies of ³He, radioactive products from copper have been observed to be various isotopes of Cu, Zn and Ga. Table I shows possible interferences of radioactivities from copper nuclei irradiated by 10-MeV ³He ions. These nuclides are positron emitters; the intensity of their 0.51-MeV annihilation radiation can be many times higher than that from ¹⁸F. Radioactives whose half-lives are either much shorter or much longer than that of ¹⁸F do not interfere and are therefore excluded. To test the effective separation of small amounts of

 18 F from these interfering activities and a possible analytical determination of trace oxygen, "electrolytic pure" copper foils were irradiated at various 3 He energies. Isotopes of Cu, Zn and Ga were removed from 18 F by chemical separation, <u>i.e.</u>, by converting them to insoluble hydroxides and recovering 18 F as 18 F₂.

<u>Calculation of Oxygen Content</u>. In this experiment, two methods for calculating oxygen in thin copper foil have been used: one is an <u>absolute</u> method, and the other is a <u>comparative</u> method. The <u>absolute</u> method calculation is given by:

$$\underline{\mathbf{D}}_{o} = \underline{\mathbf{n}} \ \underline{\mathbf{I}} \ \underline{\sigma} \ [1 - \exp(-0.693 \ \underline{\mathbf{t}}/\underline{\mathbf{T}}_{1/2})] \tag{1}$$

where

 $\frac{D}{O}$ = disintegration rate of product at the end of bombardment, in dis/min,

 $\underline{\mathbf{n}}$ = number of target atoms per sq. cm. of the nuclide being determined (16 0),

 \underline{I} = average beam intensity, in incident ³He ions per minute (1 μA ³He²⁺ = 1.873 × 10¹⁴ ³He²⁺ per minute),

 $\underline{\sigma}$ = cross section for the reaction in cm² per target atom,

 \underline{t} = length of bombardment, in minutes,

 $\frac{T}{1/2}$ = half life of product nuclide, in minutes.

When both "unknown" and standard are placed in the same stack, in the comparative method in which both are bombarded in the same irradiation and are counted under identical condition, Eq. (1) reduces to

$$\frac{A}{A} = \frac{m}{m} \times \frac{\sigma}{\sigma}$$
 (2)

Where A_x and A_s are product counting rates (^{18}F) in the unknown and standard respectively, σ_x and σ_s , are the ^{18}F production cross sections in unknown and standard taken from the $^{16}O(^3\text{He,p+n})$ excitation functions, and \underline{m}_x and \underline{m}_s are masses of the oxygen nuclei in g/cm^2 . Thus, the amount of oxygen in the sample can be computed from Eq. (1) by measuring \underline{D}_o , \underline{I} , $\underline{\sigma}$ and \underline{t} , and from Eq. (2) by comparing product activity of sample and standard with correction for any differences in cross section. The counting rates, \underline{A} , are related simply to the nuclear disintegration rates, \underline{D} , by $\underline{A/D} = \underline{ODC}$, where \underline{ODC} represents the over-all detection coefficient of a particular detector for a particular radioactive nuclide; it is independently measured.

EXPERIMENTAL

Preparation of Samples and Standards. Samples were cut from a sheet of pure electrolytic copper foil (United Mineral and Chemical Corp.). Each piece was weighed on a semimicrobalance, and degreased with acetone; average masses of copper were approximately 2 mg/cm². One set of copper foils was etched with diluted HNO₃ for a few seconds to remove surface oxide. Tantalum oxide foils prepared by anodization of pure tantalum foil were used as oxygen comparative standards. The general procedure and apparatus used for preparation of standard oxygen targets by anodic oxidation was discussed recently (16). Samples of high-purity tantalum metal, one inch in diameter, were cut from one-mil foil and degreased with acetone. The mass of oxygen incorporated into the tantalum by anodic oxidation was determined by the increase in weight of the tantalum foil. Standards of tantalum oxide were used for this experiment, having thicknesses of 0.625 to 0.654 mg/cm² oxygen.

Irradiation. The copper samples and tantalum oxide standards were arranged in a stacked-foil assembly. The tantalum oxide was covered with thin gold foils to prevent ¹⁸F recoil losses. Appropriate Al foils were placed in front of the sample assembly to degrade the beam of 30-MeV ³He to about 17 MeV. The beam energy incident on the stack of Cu foils (after passing through the standards and degraders) was 11 to 12 MeV. These targets were irradiated with ³He²⁺ ions for 10 to 11 minutes at average beam currents of 1.5 μA at the Lawrence Radiation Laboratory 88-inch cyclotron. The total charge received by the Faraday cup was measured by a calibrated integrating electrometer. Beam energies for each foil were calculated from the computer code of Steward (17).

Radiochemical Separation of ¹⁸F. After irradiation, those copper foils for chemical separation were transfered to a 15 ml centrifuge cone which contained 2 ml of 4 M HNO₃, 10.0 mg of F carrier, and about 2 mg of "scavenger" carriers--Zn²⁺ and Ga³⁺ added as nitrates. After the copper dissolved, NaOH was added carefully to adjust the solution to pH 9 where the hydroxides of Cu²⁺, Zn²⁺, and Ga³⁺ precipitate. The precipitation of hydroxides was repeated once more with a few more milligrams of scavenger carriers. The solution containing F was then adjusted to about pH 5 with an acetate buffer solution and an excess of Pb(NO₃)₂ was added to precipitate PbF₂. The lead fluoride precipitate was washed with distilled water and mounted onto filter paper with alcohol. The sample of Pb¹⁸F₂ was counted after drying for a few minutes at 165 °C. After counting was completed, the chemical yield of F was determined gravimetrically by re-precipitation of the F as PbClF.

Counting. Gamma rays from activated copper foils and chemically separated Pb 18 F₂ were measured by using NaI(Tl) and Ge(Li) detectors coupled to a multichannel analyzer and a magnetic tape unit. Because 0.51-MeV annihilation radiation was used to measure the amount of 18 F present, a pair of 3-inch × 3-inch NaI(Tl) detectors in coincidence was used for the major part of this experiment to follow the annihilation photopeak more selectively. The over-all detection coefficient for annihilation quanta in coincidence was measured with a 22 Na standard (Int. Atomic Energy Authority, Vienna) to be 4.2%. The count rate from the 0.51-MeV gamma peak was obtained by fitting a Gaussian shape to the observed gamma peak, and by calculating the area under the peak by a computer code developed in this laboratory. Decay-curve analysis

of the counting data, used to separate conveniently the count rate of the 18 F photopeak and to extrapolate it to the end of bombardment, was performed by the method of least squares using the CLSQ computer code (18). The half-life of 18 F obtained in our experiments, 110 minutes, agrees well with the value of Mahony and Markowitz, $109.7_2 \pm 0.06$ minutes (19).

RESULTS AND DISCUSSION

Interferences. Spectra of copper foils irradiated at various ³He energies (with no radiochemical separation) are displayed in Figure 2 and Figure 3. The upper spectrum in each figure indicates gamma photopeaks from isotopes of Cu and Ga present in large amounts. The predominant high-energy gamma peaks diminished as the incident energy of ³He decreased to below 6 MeV as shown in both Figure 2 and Figure 3. Figure 4 shows a comparison of two copper samples irradiated by 11.4-MeV ³He; the upper spectrum was obtained from direct counting of one of the copper foils and the lower spectrum was obtained from Pb ¹⁸F₂ which was chemically separated. The experiment demonstrates radiochemical separation even in the presence of a very high intensity of unwanted activities.

Decay curves of the 0.51-MeV photopeak from copper activated by ³He ions of various energies are plotted in Figure 5. The slope of the decay curves for ³He energies below 6 MeV corresponds to a half-life of 110 minutes with negligible interferences. As the energy of ³He is increased, the shape of the decay curve changes with the appearance of a long-life component corresponding to a half life between 9.5 and 10 hours. Analyses of the decay curves indicate that the long-life component is a mixture of 9.5-h ⁶⁶Ga and 12.8-h ⁶⁴Cu. Some ⁶¹Cu (3.3 h) was also present at ³He energies higher than 8 MeV. Elimination of interferences solely by decay-curve analysis of the annihilation gamma for the energies of ³He above 8.5 MeV was found to be inaccurate because of the large amount of interfering activity from long-life components and because of ⁶¹Cu having a half life different by less than a factor of two from ¹⁸F. As the energy of ³He decreases, the ratio of activity of ¹⁸F to

the long-life component (64 Cu + 66 Ca) increases and production of 61 Cu decreases very rapidly; ratios of 18 F to [64 Cu + 66 Ca] were approximately 2 at 8.5 MeV, 9 at 7.5 MeV and 90 at 6.3 MeV. Some cross sections for production of 18 F from 16 O are listed in Table II. The ratio of 18 F cross section at 6.3 MeV to 7.5 MeV is approximately 0.9; the relative activity, however, of 18 F to the interfering activity for these two energies is 10. Therefore, a nondestructive analysis of oxygen in copper can be measured with minimum interferences and maximum sensitivity at the incident energy of 3 He of about 6.3 MeV.

Most of the resulting nuclides, produced by activation, decay with a short half-life directly to the ground state. Interferences of this sort can be eliminated completely by delay of the start of the counting. There is one element, however, whose presence in a copper sample would produce an interference if $^{18}{\rm F}$ is to be the signal for $^{16}{\rm O}$. Fluorine, if present, would produce $^{18}{\rm F}$; via the $^{19}{\rm F}(^3{\rm He},\alpha)^{18}{\rm F}$ reaction; the cross section for the production of $^{18}{\rm F}$ from $^{19}{\rm F}$ is about 15 mb at 6.5 MeV. The $^{19}{\rm F} \longrightarrow ^{18}{\rm F}$ cross section is, however, considerably lower than the $^{16}{\rm O} \longrightarrow ^{18}{\rm F}$ cross section which is approximately 350 mb at 6.5 MeV (5). It will therefore require 24 times as much fluorine as oxygen to produce an equal amount of $^{18}{\rm F}$ at 6.5 MeV. We assume this amount to be improbable in electrolytic copper. Other elements may produce activated products having half-lives of positron emitters close to 110-min $^{18}{\rm F}$. If they are present in considerable amounts, a chemical separation would eliminate these interferences.

Results from Nondestructive Analysis. Table II shows results of eleven analyses which were obtained from four different irradiations for the nondestructive analysis of oxygen in copper foils. The results in column 5 were calculated based on tantalum oxide as the oxygen standard, and the results in column 6 were obtained by absolute calculation. Because both sample and standard were thin, sample and standard in the same stack received the same intensity of the collimated 3 He beam; only variations of the energy of the 3 He beam as it traversed the target were taken into account. The appropriate range-energy relations and excitation function were used to correct the yield for the loss in energy as the beam traverses the stack. The average analyses for 0 in Cu based on Ta_2O_5 as the oxygen comparative standard compared to the analyses based on the absolute method are 0.05L_6^{\pm} 0.003% and $0.055^{\circ}_9^{\pm}$ 0.003% by weight, respectively.

Results from Destructive Analysis. A comparison of destructive and nondestructive analysis of oxygen in copper foils is listed in Table III for the same average incident energies of $^3\mathrm{He}$. Two sets of targets made up of two identical stacks of copper foils and standards were irradiated consecutively. A chemical separation of $^{18}\mathrm{F}$ was performed for one set. Annihilation radiation from both targets was counted by using NaI(T1) in coincidence. The average chemical yield of $^{18}\mathrm{F}_2$ was approximately 50%. The results gave 0.052 $^{\pm}$ 0.009% 0 in Cu.

Sources of Error and Precision Estimate. From Table II, column 4, a standard deviation of 18 F activity from the decay-curve analysis indicates a precision of approximately \pm 1% at 5.7-to 7.5-MeV 3 He, and a rapid increase in counting error above 7.5 MeV. The uncertainty of 18 F activity is mainly

due to counting statistics and data-point scattering in the lower-energy region of the ${}^3\text{He}$; but at a higher energy of ${}^3\text{He}$, the interfering components contribute to a large portion of uncertainty in the decay-curve analysis. Other sources of error include lack of homogeneity of oxygen in the copper foils, and errors in the range-energy calculations and cross sections. In this experiment, the average from all nondestructive analyses was $0.055 \pm 0.003\%$ oxygen in copper which gave a $\pm 5\%$ relative standard deviation.

For <u>destructive</u> analysis an additional source of error results from the uncertainty of the chemical-yield determination. The average from the destructive measurements was $0.052 \pm 0.009\%$ oxygen in copper, giving a relative standard deviation of $\pm 17\%$. (The relative standard deviation here (17%) is of this magnitude mainly due to one run at 11.4 MeV not excluded from the average.)

Sensitivity Estimate. Under the following conditions, easily attainable in practice,

 $\underline{D}_{0} = 10 \text{ d/m}^{-18} \text{F} \text{ (ODC} = 50\% \text{ for positron end-window proportional counter)}$

 $\underline{I} = 10 \mu A^{3} \text{He}^{2+} \text{beam}$

 $\underline{\sigma}$ = 350 mb at ³He energy 6.5 MeV

 \underline{t} = length of bbt, one $T_{1/2}$ of 18 F, 110 minutes,

we calculate $\underline{n} = 3 \times 10^{10}$ atoms $^{16}\text{O/cm}^2$ are detected corresponding to a mass, $\underline{m} = 8 \times 10^{-13}$ grams $^{16}\text{O/cm}^2$. Therefore, in a Cu foil, for example, of thickness 2.3 mg/cm² (0.1 mil), the sensitivity would be 0.35 parts per <u>billion</u> (ppb), or \sim 29 dpm/ppb. This compares well with the sensitivity 25 dpm/ppb calculated

from the experimental data in Table II. (Modern cyclotrons achieve beam currents of 50 to 100 µA and the length of bombardment could be increased to "saturation" to gain a further division of the sensitivity by a factor of 10 to 20 to reach 0.018 to 0.035 ppb. Targets properly water-cooled-especially feasible for a Cu target-sustain this beam power.)

A practical measure of sensitivity, however, is a balancing of precision, interferences, convenience, cost, and the level of accuracy needed for a particular matrix. If a 3-inch by 3-inch NaI scintillation spectrometer is used, the over-all detection coefficient for singles counting is about 10% and normally the length of a bombardment is short compared to the half life of the desired radioactivity (herein 10 minutes for the 110-min ¹⁸F). Under these conditions, a sensitivity of 15 ppb, compared to 0.35 ppb described above, would be readily achieved.

In practice it remains a matter of speculation whether the analysis would ever have to be pushed to these low sensitivities because of the difficulty of actually producing copper that free from oxygen impurity. In any case, the potential is there.

CONCLUSIONS

In this research, we have demonstrated the method of oxygen analysis in copper by both destructive and nondestructive means. By control of the incident 3 He energy, however, we show that nondestructive analysis can be successfully carried out, eliminating the need for chemical separation. At 3 He energies of 4.7 to 6.3 MeV (Table II), the cross section for the 16 O \longrightarrow 18 F remains high, i.e., 165 to 350 mb. Because the sensitivity is very high, reduction of the beam energy eliminates Cu-induced interferences without hurting sensitivity. The depth of penetration decreases, of course.

Further, we showed that it was not necessary to use 0.51-0.51 MeV coincidence detection; direct counting of the "singles" 0.51-MeV γ with NaI was sufficient for accurate determination of the $^{18}\mathrm{F}$. In addition, at $^{3}\mathrm{He}$ energies below 6.3 MeV, the decay of the $^{18}\mathrm{F}$ gave essentially one-component decay curves enabling direct and rapid determination of the $^{18}\mathrm{F}$ without the need for elaborate computer techniques.

The key to practicality of the method remains, therefore, the availability of a ${}^3\text{He}$ accelerator. Low-energy ${}^3\text{He}$ beams (3 to 8 MeV) are attainable with small, "desk-top" cyclotrons previously discussed by Markowitz and Mahony ($\underline{5}$). Van de Graaff accelerators also may be used for about 3 to 6 MeV.

In this paper, we have demonstrated activation analyses by a "batch" or "sampling" process. With low-energy 3 He beams using ll0-min 18 F, or possibly 2.1-min 15 O from the 16 O(3 He, α) 15 O reaction, with a fixed irradiation time, a fixed delay time, a fixed counting time, and with a monitored beam, nondestructive "continuous" analyses could be developed.

Work performed under the auspices of the U.S. Atomic Energy

Commission.

Table I. Possible Interfering Nuclides from Cu Irradiated by 10-MeV $^3\mathrm{He\ Ions.}$

Target nuclide	Reaction	Product nuclide	Cross section (mb)	Half- life	Q-value (MeV)	0.511-MeV intensity (% of disinte- grations) ^a
65 _{Cu}	(³ He,2n)	66 _{Ga}	121	9.5 h	-4.8	114
63 _{Cu}	(³ He, an)	61 _{Cu}	14	3.3 h	+0.8	120
63 _{Cu}	(³ He,2p)	64 _{Cu}	26	12.8 h	+0.2	38

There are 2 annihilation radiation photons per β^{+} emitted.

Table II. Results from Nondestructive Analysis of Oxygen in Copper Foils by Various Energies of $^3\mathrm{He}$ Ion.

		Beam Intensity ∿1.5 μA A 18 _F		Length of bbt ∿10 min	n ODC = 4.2%
Copper foil	³ He Energy (MeV)	Cross section σ(mb)	A F (counts/min)	Oxygen found in Cu(% based on Ta ₂ O ₅ std.) Oxygen found in Cu(%) based on absolute method
1	3.7	55	1842 [±] 50	0.051	0.056
2	4.5	140	5089 ± 327	0.061	0.064
3 ^a	4.7	165	5322 ± 186	0.053	0.056
4	4.7	165	5473 ± 90	0.055	0.054
. 5 ^a	5.3	230	7331 ± 110	0.053	0.054
6	5.3	230	7011 ± 110	0.052	0.051
7	5.7	290	9577 ± 105	0.056	0.056
8	6.3	350	10603 ± 113	0.053	0.055
9	7•5	390	12218 ± 109	0.054	0.055
10	7.8	350	11065 ± 220	0.053	0.053
11	8.5	335	11610 ± 1205	0.060	0.061
			Average	0.054 ₆ ± 0.00	0.055 ₉ ± 0.003%

 $^{^{\}mathrm{a}}$ Samples 3 and 5 were etched with dilute HNO $_{3}$ for a few seconds.

Table III. Comparison of Destructive and Nondestructive Analyses at the Same $^3\mathrm{He}$ Energies and Identical Counting Conditions.

3He energy (MeV)	Oxygen in Cu(%) with chem. sepn.	Chemical yield (%)	Oxygen in Cu(%) without chem. sepn.
6.3	0.054	37.4	0.053
7.5	0.046	50.2	0.054
8.5	0.045	49.7	0.060
9.6	0.046	58.4	
11.4	0.069	37.4	
<u>A</u>	verage 0.052 ± 0.009%		0.056 ± 0.003%

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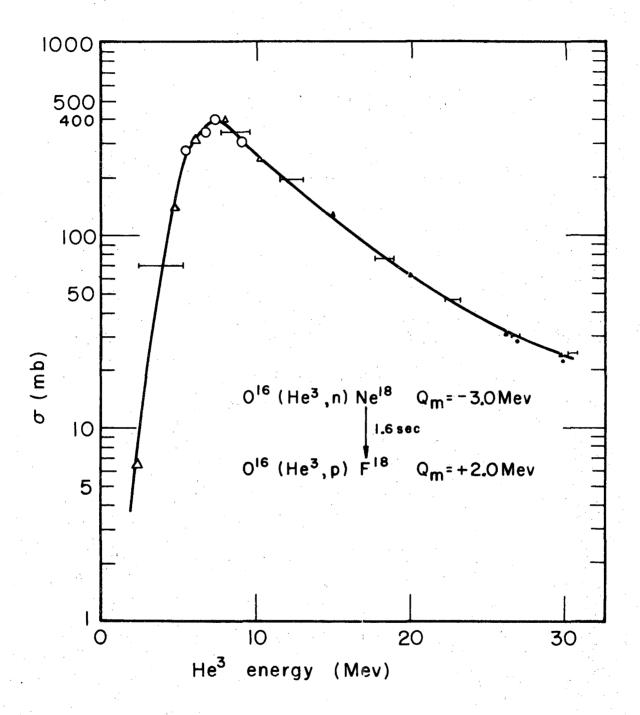
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FIGURE CAPTIONS

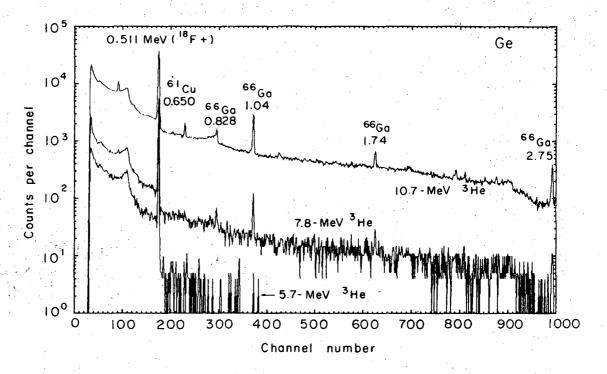
- Figure 1. Excitation function for formation of 18 F from 16 O + 3 He.
- Figure 2. Gamma-ray spectra of copper foil irradiated by 10.7, 7.8, and 5.7-MeV ³He ions: <u>Ge detector</u>, no chemical separation.
- Figure 3. Gamma-ray spectra of copper foil irradiated by 11.4, 7.5, and 6.3-MeV

 3He ions: NaI detector, no chemical separation.
- Figure 4. Comparison of gamma-ray spectrum of chemically-separated ${\rm Pb}^{18}{\rm F}_2$ with that of copper foil counted without chemical separation of ${}^{18}{\rm F}_2$. Irradiated at 11.4-MeV ${}^{3}{\rm He}$; NaI detector.
- Figure 5. Decay curves of 0.51-MeV annihilation radiation photopeak at various ^3He incident energies.



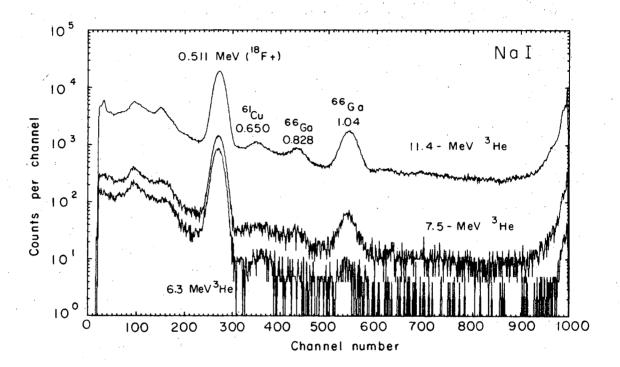
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Figure 1.



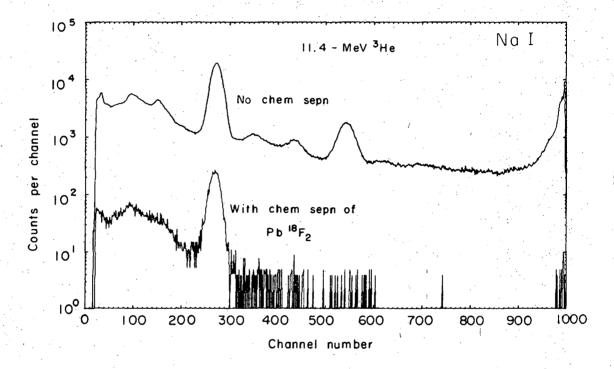
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Figure 2.



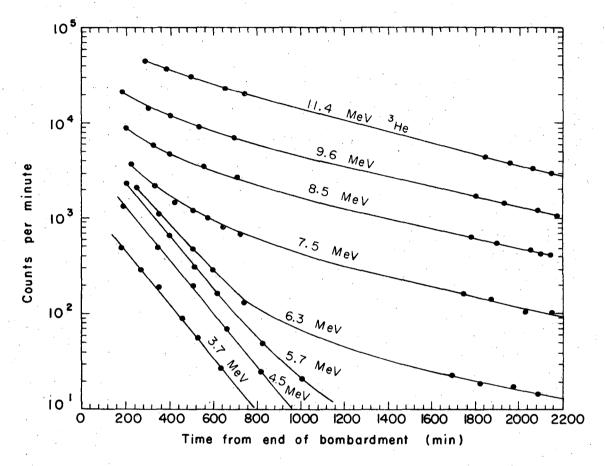
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Figure 3.



XBL 6910 - 3988

Figure 4.



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Figure 5.

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