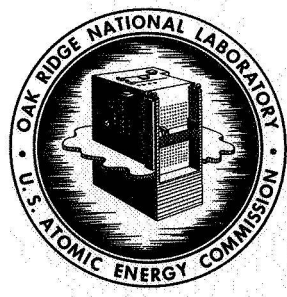


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PRODUCTION STUDY OF GADOLINIUM-153

F. N. Case
E. H. Acree
N. H. Cutshall

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ORNL-TM-2632

Contract No. W-7405-eng-26
ISOTOPES DEVELOPMENT CENTER

PRODUCTION STUDY OF GADOLINIUM-153
Prepared for NASA, Langley, Hampton, Virginia
(Interagency Agreement AEC 40-108-67, MIPR-L-1775)
Summary of Results March 1967-December 1968

F. N. Case
E. H. Acree
N. H. Cutshall

Isotopes Division

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AUGUST 1969

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ABSTRACT

Gadolinium-153 decays by electron capture, yielding predominantly ~100-keV gammas. Production methods, chemical purification, and output and gamma spectrum characteristics of fabricated sources have been studied in detail to obtain data necessary for fabrication of sources useful in the development of atmospheric density gages based on gamma backscatter measurements.

INTRODUCTION

The determination of atmospheric density by use of gamma backscatter has been studied by a number of investigators.¹ While techniques for measuring gamma backscatter from solids and gases have been demonstrated, application of the principle to specific problems requires the use of gamma sources that have specific gamma energy spectra and output intensity critically matched to the requirements of the measurement to be obtained. In the case of atmospheric density measurements, it has been calculated and experimentally determined that the gamma scatter in air from a source of 100-keV photons is related to the air density and thus provides an instantaneous readout of density by measuring the scatter intensity. Such a system appears to be adaptable to the measurement of atmosphere on other planets by passing a probe through the atmosphere.

A study funded by NASA, Langley at Hampton, Virginia, was initiated to identify radionuclides useful in atmospheric measuring devices and evaluate production methods necessary to obtain sufficient quantities for a Mars probe, as well as to evaluate source fabrication methods and techniques for calibration of the sources.

In a study to determine the optimum source of 100-keV photons for use in generating backscatter signals, 21 radionuclides were evaluated. Those radionuclides having high energy components in their radiations make it

¹N. W. Gebbie, Final Report - Mars Probe Lander Density Sensing System, NASA CR-66094 (February 1966).

necessary to increase the source shield weights required to attenuate direct radiation from the source to the detector. Of this group only three, ^{153}Gd , ^{57}Co and ^{155}Eu , warranted in-depth study; ^{153}Gd was chosen as the best from the standpoint of cost, energy characteristics, half-life, and output. This is in agreement with earlier studies¹ that indicate ^{153}Gd to be useful in atmospheric density measurements by gamma back-scatter. Since the quantity and radiochemical purity of the radionuclide required were much greater than those previously obtained, development of methods to produce multicurie quantities containing only a few ppm radiochemical contamination and to determine time cycles and costs associated with large quantity production was started. This report is a summary of the results of this development for the period March 1967 through December 1968.

PRODUCTION OF GADOLINIUM-153

Nuclear Properties

Gadolinium-153 decays by electron capture with a half-life of 241 days.² The gamma energies and percent yield are shown below:

<u>Gamma Energies</u> (keV)	<u>Percentage</u>
70	3
97	30
103	22

A gamma spectrum which was obtained with a germanium diode is shown in Fig. 1.

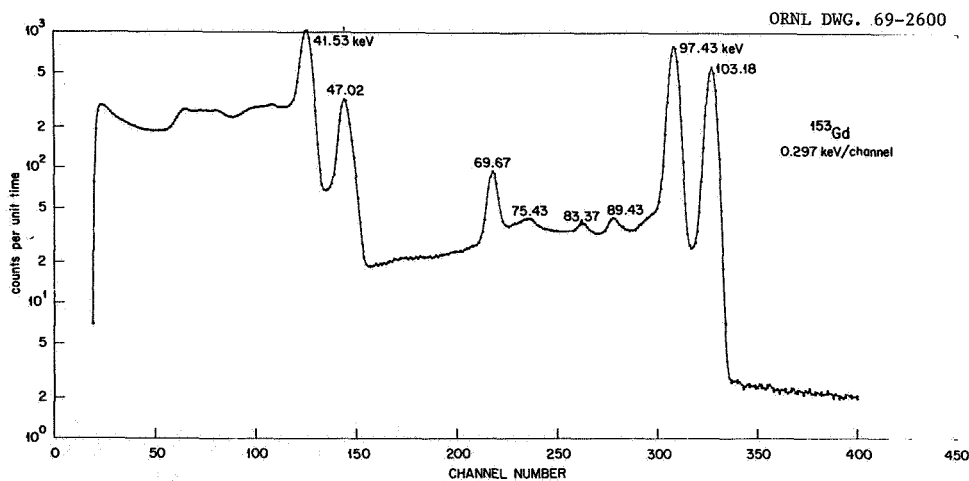


Fig. 1. Gamma Spectrum Obtained with a Germanium Diode.

²S. A. Reynolds, Oak Ridge National Laboratory, private communication, May, 1969.

Production of Gadolinium-153 from Enriched Gadolinium-152

Cross-Section Measurements. A ^{153}Gd production scheme suggested by Giannini Controls Corporation in previous work¹ involved complete conversion of ^{152}Gd (gadolinium enriched to 13% ^{152}Gd) into ^{153}Gd by neutron bombardment. However, no allowance was made for reaction of ^{153}Gd with neutrons during irradiation (target burnup). The first task of the ORNL work involved measurements of thermal neutron cross sections and resonance integrals for ^{152}Gd and ^{153}Gd . Target samples consisting of 50 μg to 3 mg of Gd_2O_3 with flux monitors were irradiated in the Oak Ridge Research Reactor (ORR). Similar samples enclosed in cadmium were also irradiated. After irradiation, the samples were analyzed by mass spectrometry and by gamma-ray spectrometry. Resonance integrals were computed from isotopic changes in cadmium-enclosed targets, while effective cross sections were computed from changes in the unshielded targets. Thermal cross sections were determined by correcting the effective cross sections for the epithermal reaction component.³

The thermal neutron cross section determined for ^{152}Gd , shown below, is in good agreement with published values:⁴

	<u>^{152}Gd</u>	<u>^{153}Gd</u>
Thermal cross section, barns	120	~10,000
Resonance integral, barns	12,000	~1,000,000
Effective cross section in ORR, barns	700	27,000

Gadolinium-153 was found to have a rather high thermal neutron cross section and an extremely high resonance integral.

The maximum obtainable specific activity is approximated by the relationship

$$(S)_{\text{max}} \approx (S)_{153} \frac{\sigma_{152}}{\sigma_{153}},$$

where

- $(S)_{153}$ = specific activity of pure ^{153}Gd ,
- σ_{152} = cross section of ^{152}Gd ,
- σ_{153} = cross section of ^{153}Gd .

Thus, although pure ^{153}Gd has a specific activity of 3500 Ci/g, only about 2.5% of this value, or roughly 90 Ci/g, can be attained using pure ^{152}Gd targets. More precise calculations indicate the maximum specific activity attainable in the ORR to be 78 Ci of ^{153}Gd per gram of ^{152}Gd . Both the thermal cross section ratio ($^{152}\text{Gd}:^{153}\text{Gd}$) and the resonance integral ratio are of the order of 10^{-2} . This similarity of resonance and thermal cross

³R. W. Stoughton and J. Halperin, Nucl. Sci. Eng. 6, 100-118 (1959).

⁴Nuclear Data Sheets, 1959-1965, p 1491.

section ratios indicates that no significant advantage will result from use of a neutron flux that is differently moderated. Therefore, other reactors will not produce a significantly higher specific activity than that obtained using the ORR. Production curves at different fluxes are shown in Fig. 2.

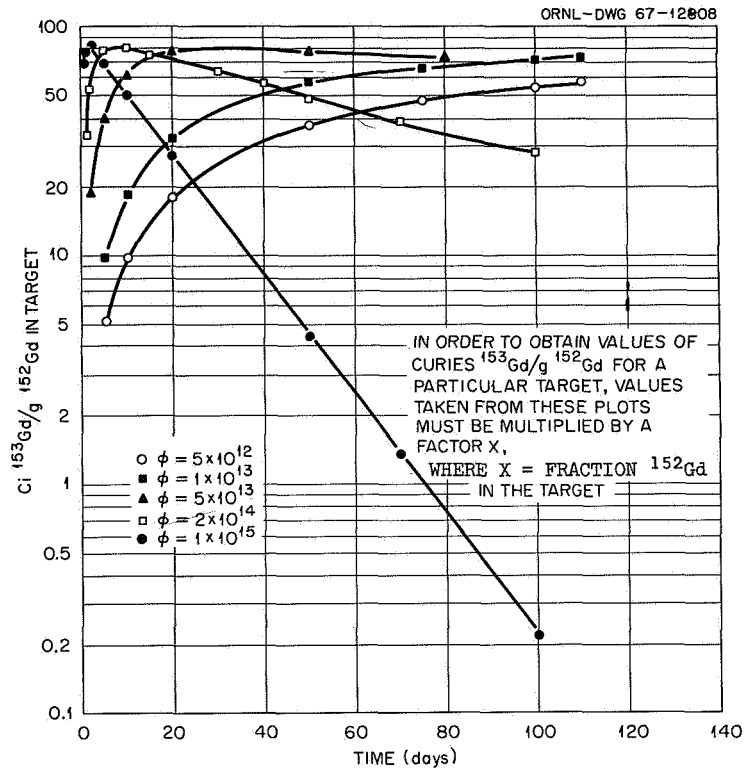


Fig. 2. Yields of ^{153}Gd from ^{152}Gd Target.

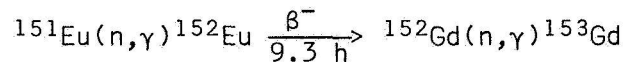
Large Sample Irradiations. Three 330-mg targets containing 13.5% ^{152}Gd (Table I) were irradiated in the ORR for 37, 56, and 93 days. This target size is comparable to that of targets which would be used in production runs. Therefore, the results of these experiments are realistic estimates of actual production yields.

Table I. Mass Analysis of Enriched ^{152}Gd Targets

Isotope Mass No.	Percent Abundance		Thermal Neutron Cross Section (barns)
	Natural	Enriched	
152	0.20	13.5	120
154	2.15	7.06	
155	14.73	23.77	58,000
156	20.47	20.97	
157	15.68	11.52	240,000
158	24.87	13.96	3.4
160	21.90	9.18	0.8

Alternative Production Method

Gadolinium-153 can also be produced by neutron bombardment of ^{151}Eu . The reaction sequence is



The computed maximum yield in a flux of 2×10^{14} neutrons/cm²/sec is 17.6 Ci per gram of ^{151}Eu (see Appendix B). If natural europium (47.82% ^{151}Eu) is used, the yield is calculated to be 8.5 Ci of ^{153}Gd per gram of target. Irradiation of natural europium also produces long-lived ^{152}Eu and ^{154}Eu , which must be separated from gadolinium. After separation, the computed specific activity of ^{153}Gd is 78 Ci per gram of gadolinium.

Since large quantities of ^{152}Eu and ^{154}Eu are produced, europium must be chemically separated from the ^{153}Gd . Two separation methods, coprecipitation and electrochemical, were investigated. The coprecipitation technique, which consists of reducing the Eu^{+3} to Eu^{+2} with a Jones reductor and coprecipitating the Eu^{+2} with Sr^{+2} as the sulfate, does not adequately separate europium from gadolinium. An 80% separation was the best that could be obtained. The electrochemical technique has proved to be much better than the coprecipitation technique, and a separation factor as high as 2000 has been obtained in the laboratory. The system is simple and requires very little equipment (Fig. 3).

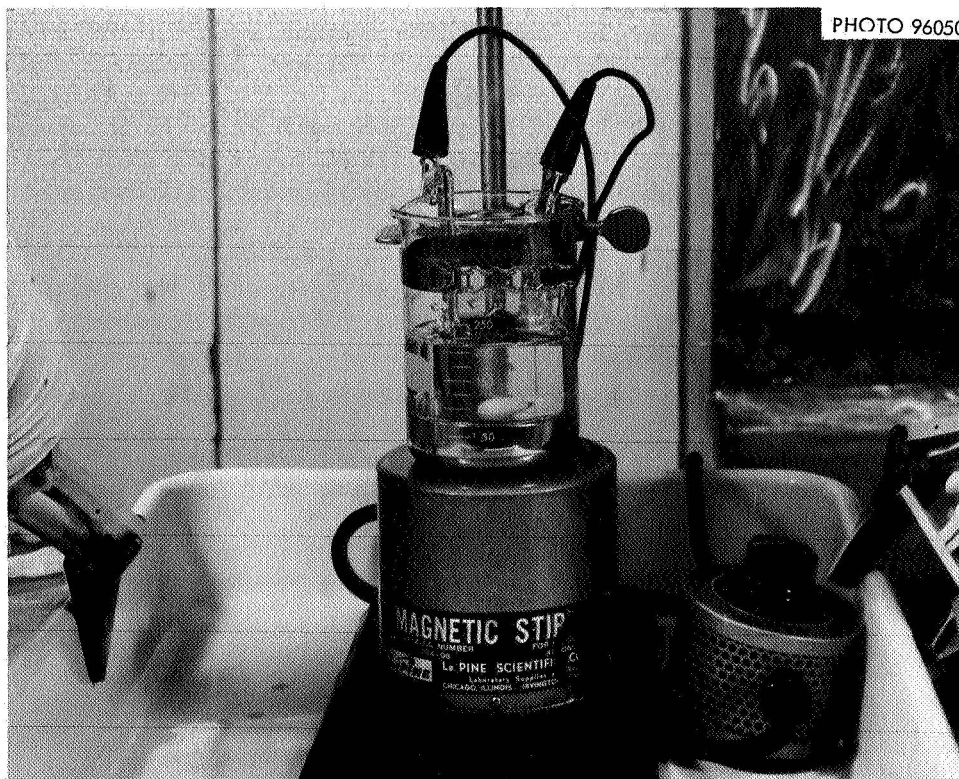
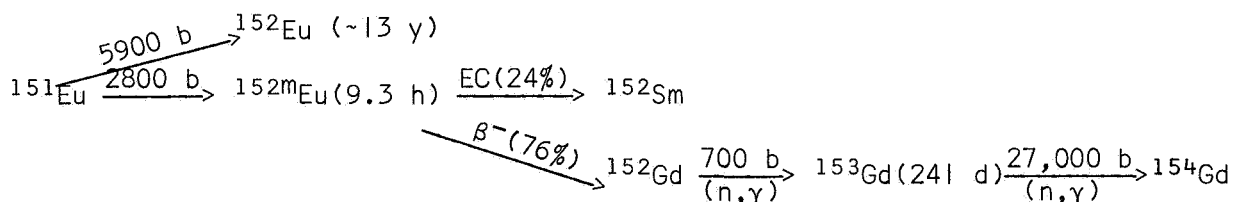


Fig. 3. Shielded Cell for ^{153}Gd Purifications.

The rare earth is dissolved and converted to an acetate, which is mixed with a saturated solution of citric acid and adjusted to pH 10 with LiOH. This solution is added to a beaker containing mercury. Contact to the mercury cathode is made with a platinum wire passed through a glass tube in such a manner that solution does not come in contact with the wire. A piece of platinum foil, 3 to 5 cm² in area, is used as the anode. A current density of ~2 ma/cm² is applied for 3 to 5 hr during electrolysis, the europium is deposited in the Li-Hg amalgam, and the gadolinium remains in solution. The electrolysis solution is decanted and acidified to ~pH 1 with HNO₃ acid. After the gadolinium is precipitated with ammonium oxalate and fired to the oxide, it is ready to be recycled through a second purification. To obtain the required radiochemical purity four recycles are necessary. This technique has been applied to a 100-Ci batch of ¹⁵³Gd, and after four separations the ¹⁵³Gd contains only 19 ppm radiochemical* impurities. Since only ¹⁵²Gd is produced from ¹⁵²Eu, ¹⁵⁶Eu and ¹⁶⁰Tb are not present in the product as would be the case if the heavier isotopes of gadolinium were present. Therefore, the use of ¹⁵¹Eu eliminates the contamination problem that one encounters if enriched ¹⁵²Gd is irradiated. This production technique is summarized in the following scheme:

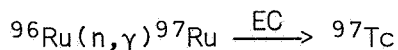


ALTERNATIVE ISOTOPES

Initial Evaluation

In cooperation with Research Triangle Institute, ORNL has reviewed possible alternatives for ¹⁵³Gd in the Mars Atmosphere Density Probe source. Research Triangle Institute selected the following nuclides for further study: ⁵⁷Co, ^{91m}Nb, ¹⁰¹Rh, ¹⁵¹Gd, ¹⁶⁸Tm, ¹⁷³Lu, ¹⁷⁴Lu, ¹⁹⁵Au, ^{97m}Tc, ⁹³Mo, ^{113m}Cd, ¹⁸⁸W, ¹³⁹Ce, ^{123m}Te, ¹²⁵Te, ^{127m}Te, ¹⁷⁰Tm, ¹⁵⁵Eu, and ¹⁰⁹Cd. The first eight nuclides are neutron deficient and can be produced only by charged-particle reactions. Of these, only ⁵⁷Co can be produced in sufficient quantity to warrant further consideration. Most of the remaining nuclides can be eliminated for the following reasons:

Technetium-97m might be produced by the reaction series



However, only 0.04% of ⁹⁷Ru decays to the desired isomeric state. The product would contain a large amount of 2.6-million-year ⁹⁷Tc and the ^{97m}Tc specific activity would be too low.

*19 Ci of other radionuclides per 10⁶ Ci ¹⁵³Gd.

Molybdenum-93 has a reported half-life of about 10,000 y; thus, the specific activity of the pure isotope is about 0.4 Ci/g.

Cadmium-113m has a 14-y half-life and only 0.1% yield of the desired gamma-ray. The maximum gamma activity is about 2 Ci/g.

Tungsten-188 decays to short-lived ^{188}Re , which emits high-energy gamma photons.

Cerium-139 could not be produced free of ^{141}Ce and ^{144}Ce .

The three isotopes of tellurium, ^{123m}Te , ^{125}Te , and ^{127m}Te , could be produced as a complex mixture; however, an encapsulated source consisting of this material would be extremely difficult to calibrate.

Thulium-170 has only a 3% yield of useful photons. It also emits beta particles up to 1 MeV in energy. The resultant bremsstrahlung radiation eliminates this isotope from further consideration.

Records of ^{109}Cd production show that the highest specific activity previously attained was only 3 Ci/g. Furthermore, the yield of useful photons is only 4%. Only ^{57}Co and ^{155}Eu remain from the original list.

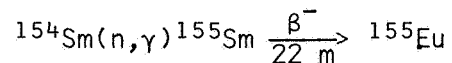
Cobalt-57 Production Feasibility

Cobalt-57 can be produced in the ORNL 86-Inch Cyclotron at about 25 mCi/beam-hr. Since the Mars Atmosphere Density Sensor source would require 50 Ci upon delivery, about 2300 hr, or 94 days, of continuous beam time would be needed.

Small quantities of ^{65}Zn are present in cyclotron-produced ^{57}Co but this impurity can easily be removed. Cobalt-57 emits a 700-keV photon with 0.2% yield (see Appendix C). Shielding to protect the Mars Atmosphere Density Sensor detector from this radiation may exceed acceptable weight limits.⁵

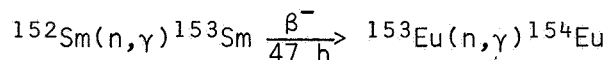
Europium-155 Production Feasibility

The best method for producing ^{155}Eu involves irradiation of enriched ^{154}Sm (see Appendix D for nuclear properties):



⁵R. L. Ely, Optimization Study of the Gamma-Ray Scattering Technique for Measuring Atmosphere Density, Source Study Report, Task 4, Contract Number NAS1-7046, Research Triangle Institute (1967).

The most serious drawback to this scheme is the simultaneous production of ^{154}Eu :



Since the saturation yield of ^{155}Eu is only 0.5 Ci per gram of ^{154}Sm (see Appendix E), chemical separation of europium from the target and target recycling would be necessary. The production sequence would be the following:

1. irradiate enriched ^{154}Sm ,
2. allow 47-h ^{153}Sm to decay,
3. separate europium from samarium,
4. **reirradiate samarium**,
5. allow 15-d ^{156}Eu in europium fraction to decay,
6. combine subsequent europium fractions,
7. fabricate source.

The ratio of $^{154}\text{Eu}:^{155}\text{Eu}$ can be minimized by use of targets with low $^{152}\text{Sm}:^{154}\text{Sm}$ ratio, short irradiation time, and frequent target reprocessing to remove ^{153}Eu (see Appendix F).

Enriched ^{154}Sm containing 98.5% ^{154}Sm and 0.5 to 1.25% ^{152}Sm is expected to be available. Irradiation of this material at 2×10^{14} neutrons/cm²/sec for 2 days should yield 0.17 Ci of ^{155}Eu per gram of samarium. The computed $^{154}\text{Eu}:^{155}\text{Eu}$ ratio would be 3 to 8×10^{-4} Ci/Ci. Although shorter irradiation times would yield a lower $^{154}\text{Eu}:^{155}\text{Eu}$ ratio, the yield of ^{155}Eu would be impracticably low.

Chemical Separations

Two of the above production methods require separation of adjacent lanthanide elements. Producing ^{155}Eu from enriched ^{154}Sm requires Sm-Eu separation. Although ion-exchange chromatography can be used to separate adjacent lanthanides, the procedures are tedious. Also, this method does not provide adequate removal of traces of one element from large quantities of the adjacent element. Furthermore, the chemistry of these three elements is sufficiently different to allow use of other methods. Two methods were tested: coprecipitation and electrochemical.

Coprecipitation Method. The Eu-Sm (Eu-Gd) mixture is dissolved in dilute hydrochloric acid solution containing Sr^{2+} . This solution is passed through a column of amalgamated zinc (Jones reductor) into sulfuric acid solution. In the column Eu(III) is reduced to Eu(II), which coprecipitates with SrSO_4 upon entering the sulfuric acid solution. Samarium (gadolinium) does not precipitate under these conditions. Because the best recovery attained for europium was only 80% per cycle, the method was abandoned.

Electrochemical Method.⁶ The best separation method we have found involves electrolysis of europium at a lithium amalgam electrode from lithium-acetate-lithium citrate solution. The Eu:Sm ratio in the product of one cycle is about 100 times the Eu:Sm ratio in the starting solution. This degree of separation is adequate for recovering ¹⁵⁵Eu from irradiated ¹⁵⁴Sm targets.

SOURCE FABRICATION

Specific Activity and Source Output. Output from a flat source of a given area is a function of specific activity and total activity. Where the area is limited and the specific activity is low, self-absorption attenuates output. For the output of monoenergetic primary photons the required source activity is given by

$$A = \frac{-SR}{\mu} \ln \left(1 - \frac{i\mu}{SYR} \right),$$

where

- A = required source input, in Ci,
- S = specific activity, Ci/g,
- R = source area, cm²,
- Y = fractional yield of useful photons,
- μ = mass absorption coefficient, cm²/g,
- i = required output, expressed as "gamma curies."

The required output of the Mars Atmosphere Density Sensor source is 20 gamma curies or 7.4×10^{11} photons/sec at Mars, and the proposed area is 10 cm² (ref. 1). For this source, specific activity is critically important in the range of 20 to 50 Ci/g. At lower specific activity, self-absorption makes it impossible to attain the required output; at higher specific activity self-absorption will be slight (see Fig. 4).

The specific activity of ¹⁵³Gd produced from currently available ¹⁵²Gd enriched to 13.5% will not be high enough for use in the Mars Atmosphere Density Sensor. While a limited quantity of more highly enriched ¹⁵²Gd is available from which approximately 130 Ci of ¹⁵³Gd at 23 Ci/g specific activity could be produced, the output of the resultant source would be only 15 gamma curies at Mars. No allowance has been made for losses during chemical processing or source fabrication. Therefore, we do not believe that production of ¹⁵³Gd from enriched ¹⁵²Gd currently available is feasible for the Mars Atmosphere Density Sensor. To verify the validity of the source output calculations, five small sources of ¹⁵³Gd were prepared and the gamma outputs measured with a 3- by 3-in. NaI crystal. These sources contained the same quantity of rare-earth oxide and ¹⁵³Gd, but the source area was varied from 10 to 18 cm². The results of this study (Fig. 5) indicate good agreement, 3 to 13% deviation, between calculated and measured outputs. The photopeaks were assumed to be 100 keV (two photopeaks exist, one at 97.43 keV and one at 103.18 keV), and an absorption coefficient of 3.08 cm²/g was used for gadolinium and 0.155 cm²/g used for O₂.

⁶E. I. Onstott, J. Amer. Chem. Soc. 77, 2129-32 (1955); 78, 2027-76 (1956).

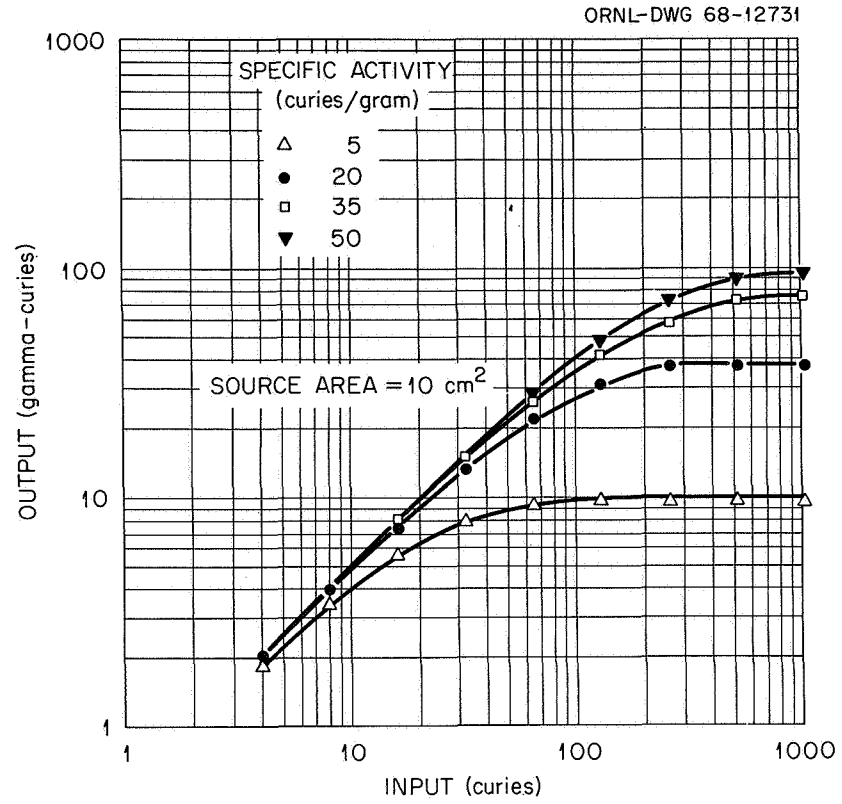


Fig. 4. Self-Absorption of 100-keV Gammas from ¹⁵³Gd.

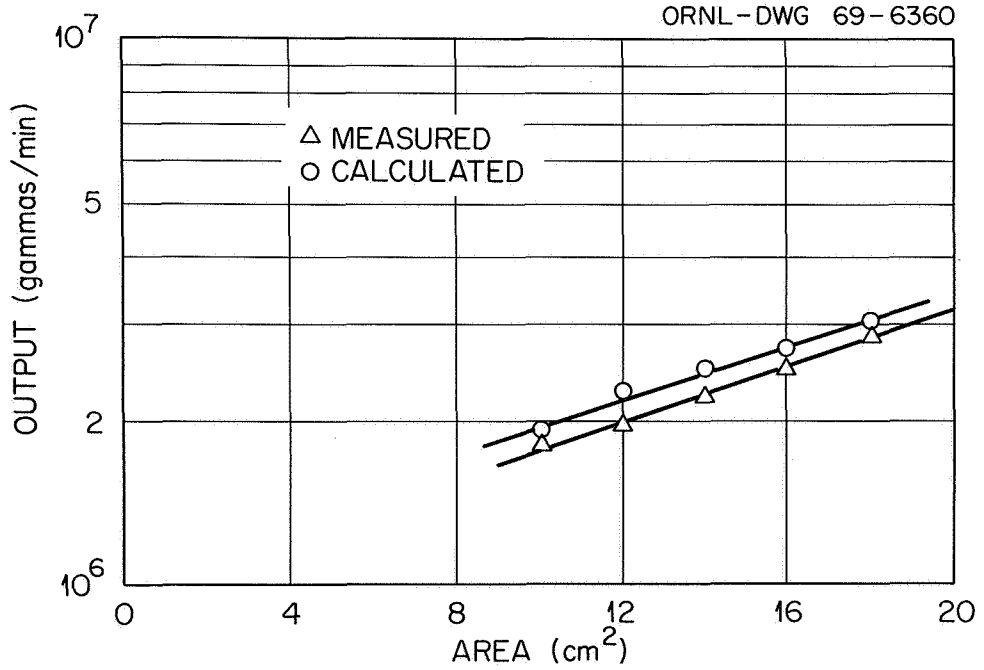


Fig. 5. Comparison of Measured and Calculated Gamma Output from Flat Plate ¹⁵³Gd Sources.



Fig. 6. Flat Plate ^{153}Gd Source and Shield.

Source Capsule. The source capsule for use in the Mars Atmosphere Density Sensor gage is type 304 stainless steel with a wall thickness of 0.010 in. The outer dimensions are $4 \frac{5}{16}$ in. long, 0.415 in. wide, and 0.105 in. thick. A $\frac{3}{8}$ -in. plug is welded in each end for closures (see Fig. 6).

1- and 5-Ci Sources. Two sources have been fabricated and shipped to the National Aeronautics and Space Administration (NASA) contractor responsible for designing a prototype sensor. One source contained 1132 mCi of ^{153}Gd as of November 26, 1968, distributed in 3.139 g of material consisting of Eu_2O_3 and Gd_2O_3 . The second source contained 5.5 Ci of ^{153}Gd in 3.15 g of the rare-earth oxides. Both sources were prepared by simply compacting the powder into the capsule. The powder was difficult to load into the capsules and small losses occurred. Pressing rectangular pellets from the oxide for loading into the capsule may overcome this difficulty.

A third source, containing approximately 900 mCi of ^{153}Gd , was fabricated and is being retained at ORNL for comparison purposes.

Table 3. Distribution of ^{153}Gd in Source Capsule

Position	Gamma Output ^a (Counts/min)	
	5-Ci Source	1-Ci Source
1	907	391
2	16,781	806
3	19,299	2,441
4	20,237	2,056
5	18,104	2,259
6	18,380	2,053
7	21,579	2,424
8	19,367	3,526
9	17,508	4,263
10	18,682	4,157
11	17,486	4,004
12	14,312	4,118
13	13,109	3,741
14	14,253	3,195
15	12,978	2,457
16	5,256	965
17	931	288

^a Measured through a 1/16-in. collimator at 1/4-in. intervals.

Uniformity Measurements. In order to maximize the gamma output, it is necessary to achieve a uniform distribution of the ^{153}Gd oxide over the length of the source capsule. Measurements were made on 1- and 5-Ci sources to determine gamma output at 1/4-in. increments. The results are shown in Table 3.

Output Measurements. The three sources were measured with an "R" meter to establish a relationship between the three sources as to gamma output. The results of these measurements are given below:

Source	Radiation (R/hr)	
	5.5 inches	11 inches
1132 mCi	1.60	0.380
5500 mCi	7.50	2.08
~900 mCi	1.36	0.32

Figure 7 depicts the physical arrangement of the sources and the "R" meter during the measurements. The numbers presented are the arithmetic mean values of several measurements.

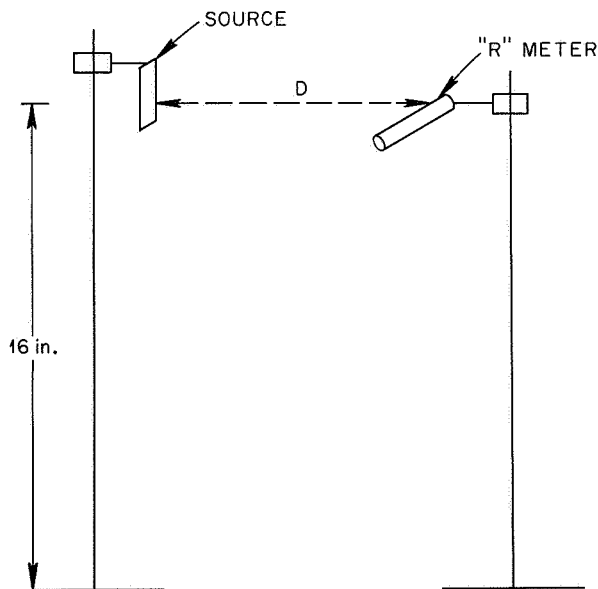


Fig. 7. Physical Arrangement of ^{153}Gd Source and "R" Meter.

There is a difference of 12% between the ratio of the input activities and the ratio of the gamma outputs. This incorporates a number of possible errors and losses and indicates that improved techniques for evaluating relative gamma output from large sources must be established before backscatter calibration data can accurately be scaled to a flight model source.

SUMMARY OF FUTURE WORK

The technique for producing ^{153}Gd by irradiating ^{152}Eu has been established and can potentially produce large quantities of high purity material. A number of small samples (1 to 5 g) will be irradiated to check yields and obtain

data relating to flux depression. Source output measurements will be continued, with emphasis on developing a measurement technique that will permit direct measurement of the 100-keV gammas. The source fabrication technique will be investigated further in an attempt to simplify the loading of the capsules and improve the distribution of ^{153}Gd in the source to yield greater uniformity over the length of the capsule.

CONCLUSIONS

Gadolinium-153 can be prepared using ^{151}Eu target in sufficient quantity and radiochemical purity to prepare sources useful in atmosphere density probes. Additional work is necessary to obtain precise calibration measurements and backscatter measurements in various gas pressures. Full-scale demonstration of the process is also necessary to determine costs and time cycles to meet future needs in space probe applications.

RELATED APPLICATIONS

As a result of this investigation sufficient quantities of ^{153}Gd have become available for other important applications, and evaluation of these applications is proceeding concurrently with the prime work objective.

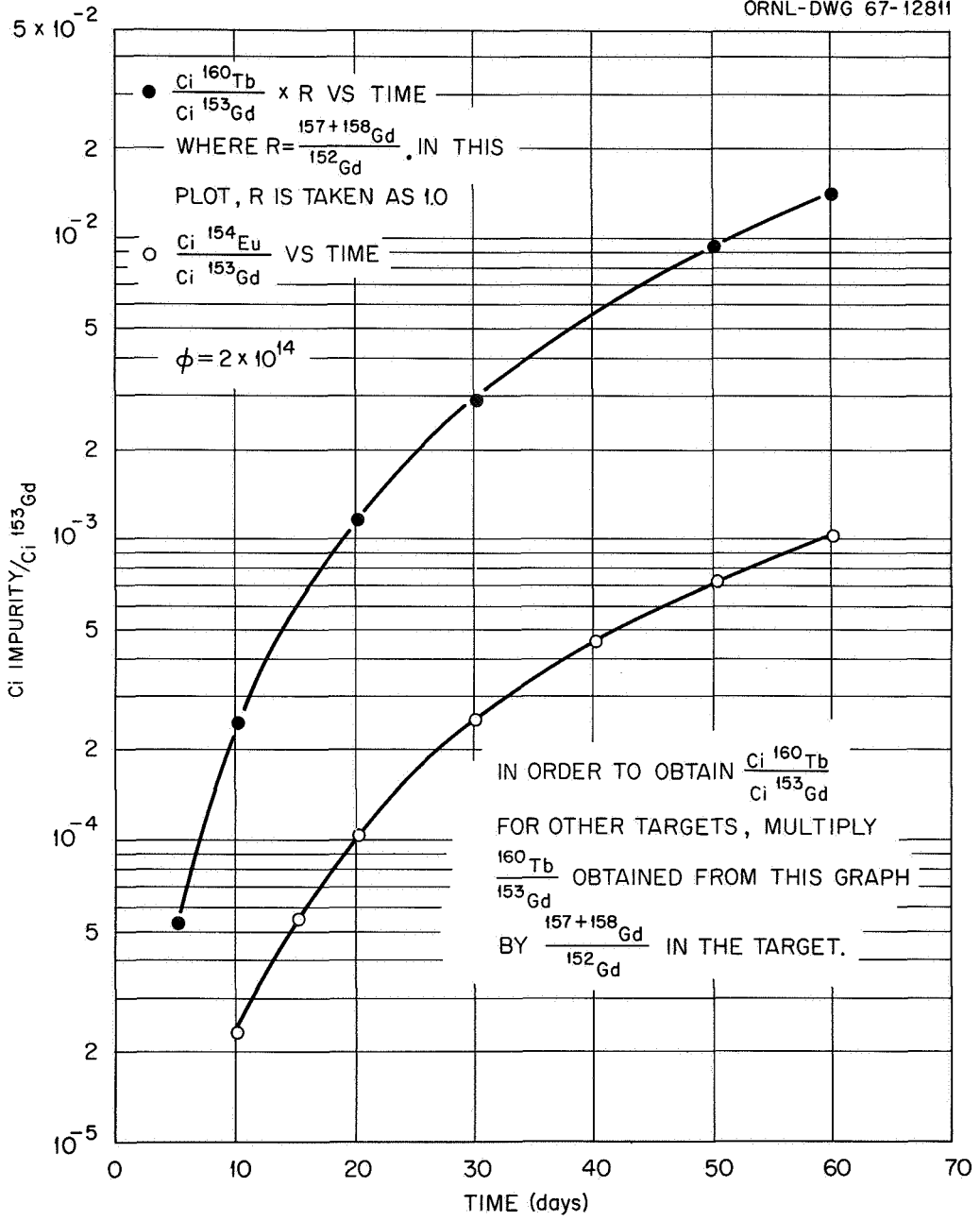
While these applications are funded by AEC, Division of Isotopes Development, the joint effort has provided for a nonduplicating type of inter-agency development program of high productivity in attainment of program objectives.

Gadolinium-153 has been evaluated for use as a source in radiographic inspection of light-weight materials in the automobile industry; applications in thickness gaging by gamma backscatter in solids are also under investigation for use in routine component production.

Sources of low-energy gamma rays utilizing ^{153}Gd are under investigation for medical diagnosis of lung disease. Such sources may be useful in reducing the cost and total-body radiation dose now experienced in lung scanning.

APPENDIX A

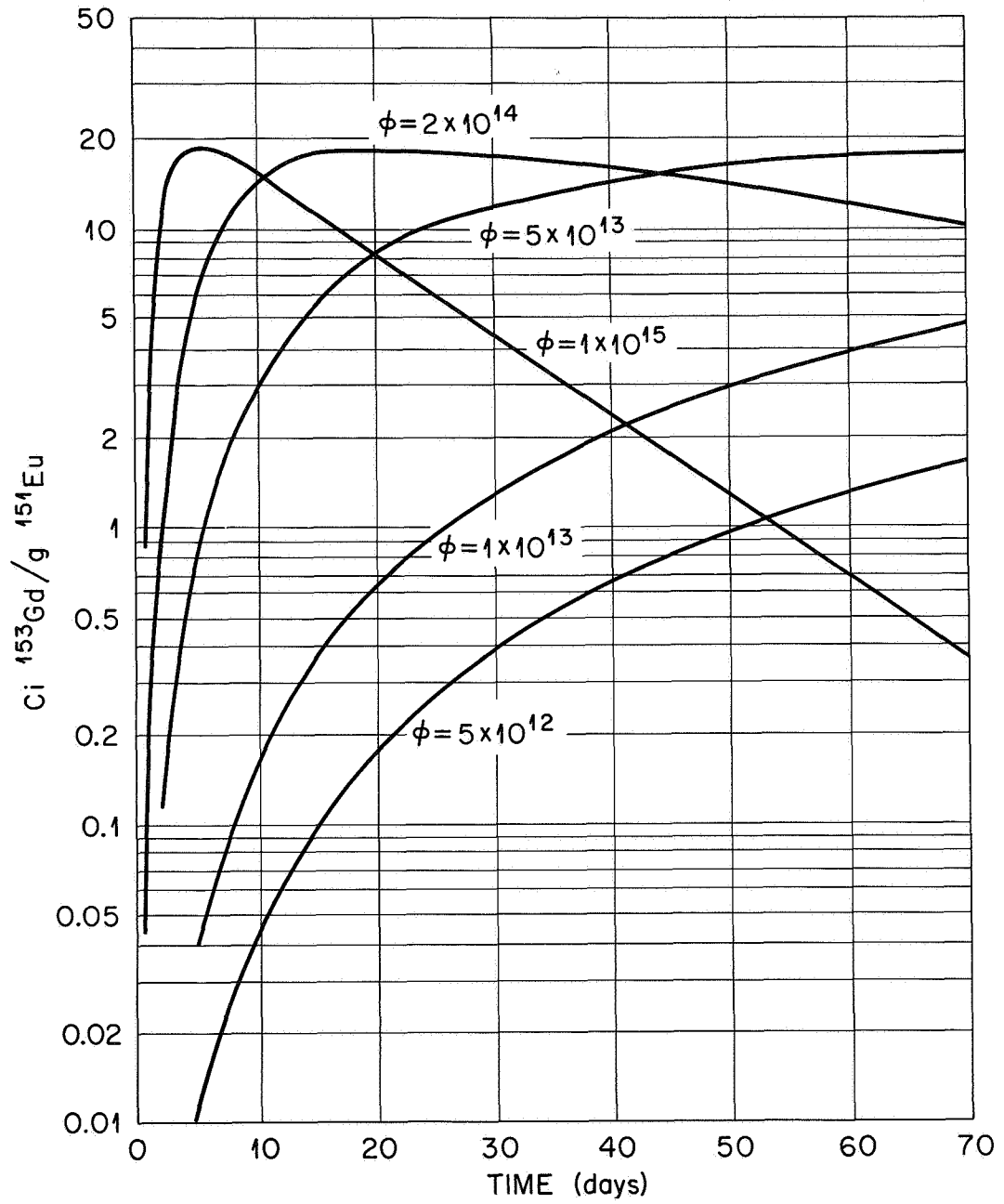
ORNL-DWG 67-12811



Production of ^{160}Tb Contaminant in ^{153}Gd .

APPENDIX B

ORNL-DWG 69-6362

Production of ^{153}Gd from ^{151}Eu .

APPENDIX C

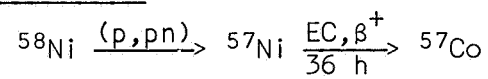
Nuclear Properties of ^{57}Co ^{57}Co Half-Life

$$T_{1/2} = 270 \text{ d}$$

Type of Decay

Electron Capture - 100%

Gamma Energies (keV)	Percent Yield
14	8.2
122	88.8
136	8.8
708	0.2

Production

Production Rate (86-Inch ORNL Cyclotron)

25 mCi/beam-hour

APPENDIX D

Nuclear Properties of ^{155}Eu ^{155}Eu Half-Life

$$T_{1/2} = 1.81 \text{ y}^*$$

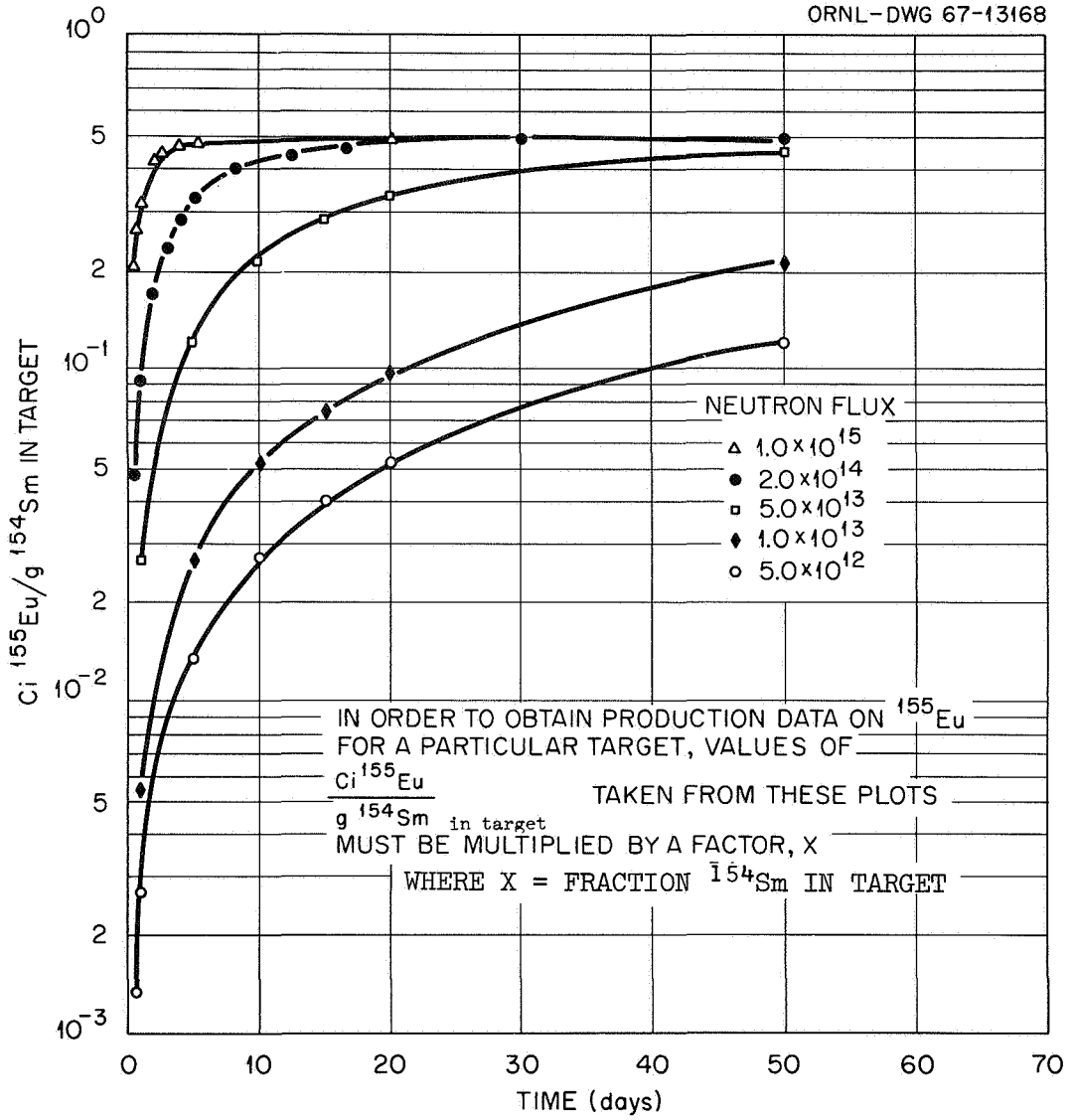
Type of Decay	Decay Energies (keV)	Percent Abundance
β^-	140	43
	158	32
	187	10
	247	15

Gamma Energies (keV)	Percent Yield
60	1.9
87	31.7
105	20.2
40 (X-ray)	25

*Recent data indicate this value may be 5 y.

APPENDIX E

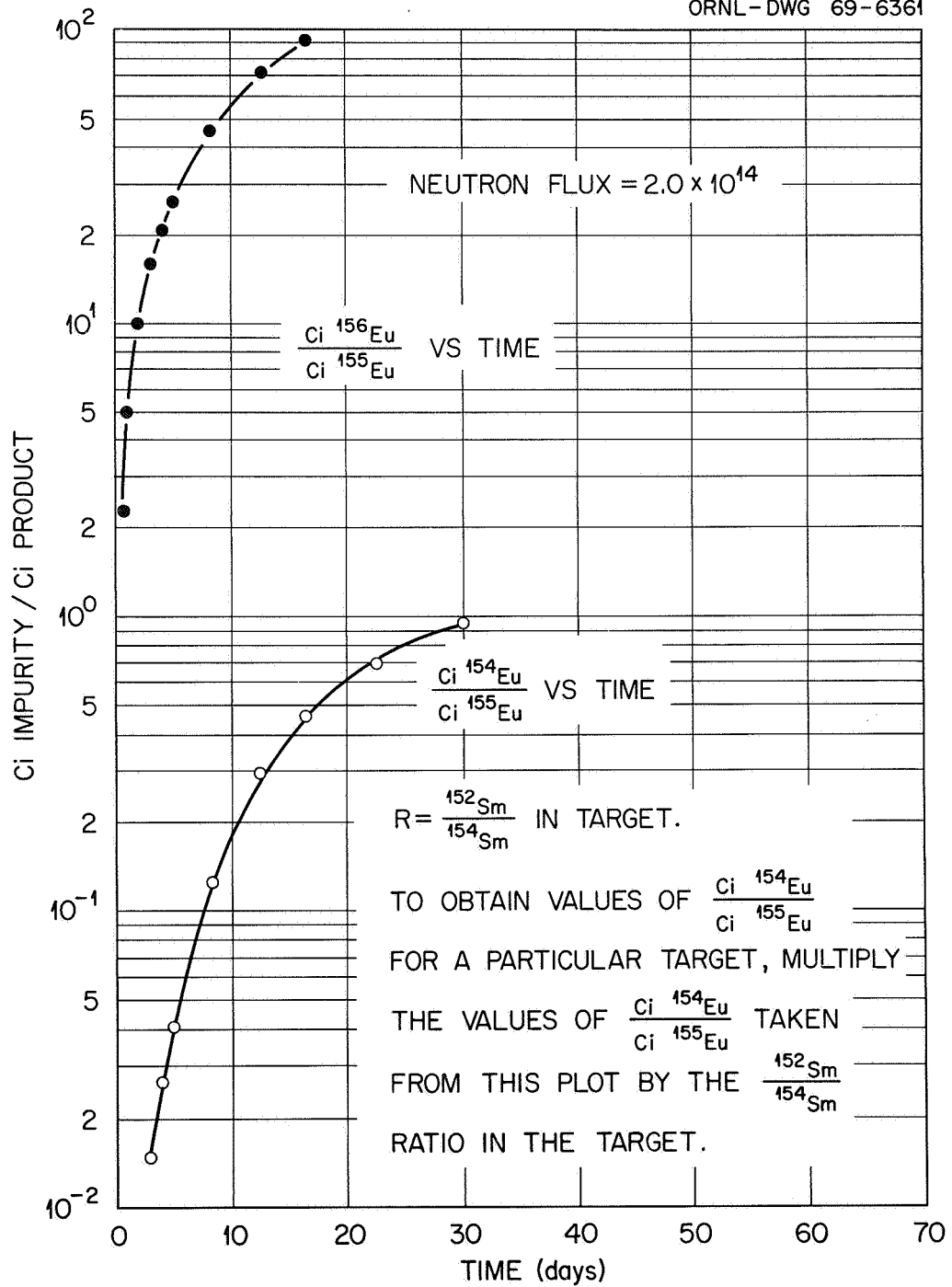
ORNL-DWG 67-13168



Production of ^{155}Eu from ^{154}Sm .

APPENDIX F

ORNL-DWG 69-6361



Production of Contaminants in ^{155}Eu .

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