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# PRELIMINARY INVESTIGATION OF THE FISSION PRODUCT RETENTION ABILITY OF CERMET COMPACTS

W. E. Moody, Jr.
A. J. Taylor
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# PRELIMINARY INVESTIGATION OF THE FISSION PRODUCT RETENTION ABILITY OF CERMET COMPACTS

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J R Johnson

Ceramic Laboratory,
Metallurgy Division

## ABSTRACT

A relatively simple comparative method for measuring fission product escape from solid fuel elements has been devised Although the method has limitations, it serves to "screen' various materials as to their relative ability to retain volatile or gaseous fission products The Xe, I and Te which were collected from the ceramic specimens employed in this study are probably representative of the fission products which are most likely to escape On this premise it is shown that between 900° C and 1000° C about 0 001% to 0 01% of the gaseous or volatile fission products are likely to escape from nonporous ceramics

In addition to the above fission products, others collected from tests made on loose $UO_2$ powder 'standards" included Mo, Ba, Zr, Sr, and rare earths

## Introduction

In order to prevent contamination of the machinery and reduce the shielding requirements in reactor power systems, fission product leakage from the fuel elements should be kept at a minimum Some means to evaluate the leakage from various kinds of solid fuel elements is thus necessary The most desirable test for a fuel element would be an in-pile loop experiment, however, a less expensive screening test has been devised which gives some information about relative fission product retention in the fuel elements A number of ceramics have been examined, comparing the fission product losses from "sandwich"-type specimens with losses from $UO_2$ powder "standards"

The test has certain limitations The in-pile exposure producing the fission products is low so that there is virtually no burn up or radiation damage Not all of the fission products are generated in sufficient quantity to permit detection in the final radiochemical analyses Short lived isotopes are not detected inasmuch as two to three days elapse from the time of removal from the pile to the time of analysis The isotopes which are solely beta emitters would not be detected in certain phases of the experiment Nevertheless, a considerable amount of useful information has been obtained At 1000° C the leakage of certain volatile fission products from nonporous cermets is reduced by $10^4$ to $10^5$ over the loss from loose $UO_2$ powder under the conditions of this test The relatively good check between the amount of fission products collected and measured and the amount calculated to have been generated in the $UO_2$ samples is particularly interesting For example, about 68% of $I^{133}$ is

is accounted for Inasmuch as these were experiments where some of the details were still being worked out and some fabrication features were imperfect, the reported results are probably somewhat conservative

Experimental Procedure

A schematic representation of the experimental procedure is shown in Figure 1 The ceramic specimens were composed of cermet type bodies with a uranium compound comprising the central portion in a sandwich type construction The compositions of the bodies and uranium compounds are given in Table III The structure of the specimens can be seen from the radioautograph (Figure 4)

The specimens were irradiated in the X-10 Graphite Reactor for periods of 7 days in a flux of $5 \times 10^{11}$ n/cm$^2$/sec On the morning of the 8th day they were sealed in silica glass containers (Fig 3) and heated for 24 hours The experiments reported here involved heating at three different temperatures, 800° C, 900° C and 1000° C On the 9th day the stem sections of the glass container were immersed in liquid nitrogen for 5 hours after which the stems were sealed off and removed for radiochemical analyses A scintillation counter, ionization chamber and gamma spectrometer were used in the analyses After examinations were made of the sealed capsules, they were broken, and studied further The aluminum-silicate "wool" used to enclose the specimens was also examined in some cases Surface Np was found by washing the specimens in acid, basic, and neutral solutions which were analyzed radiochemically

In order to make the test a valid comparison type, "standard' specimens were prepared These are identified as G-33-1 and G-224-1

A small silica glass ring was filled with aluminum silicate wool having in its center a small amount of very fine $UO_2$ powder (-325 mesh) G-33-1 contained 3 3 mg $UO_2$ while G-224-1 contained 22 4 mg Thus it was assumed that the volatile fission products could readily escape from the relatively loose wool compact

The specimens A, B, C and D were 1 1 cm diameter by 0 1 cm thick with the covering layers and the fuel portion of the "sandwich" having about equal thickness ($\sim$ 0 03 cm) The E specimens were 1 4 cm diameter and 0 4 cm thick, the covering layers being about 0 1 cm

The specimens were placed in the pile at 9 00 A M on Mondays and removed at 9 00 A M on the following Mondays An arbitrary time, 9 30 A M Wednesdays, was selected as the reference time for the radiochemical analyses in each experiment

Discussion of Results

The relatively short in-pile exposure was sufficient to generate in experimentally useful quantities only those fission products in the bimodal peaks of the fission yield spectrum Among those elements generated, however, are Xe, I and Te, which are representative of those isotopes most likely to diffuse through and escape from solid fuel elements at high temperatures Thus the results may be interpreted to indicate, at least to an order of magnitude, the relative merits of various specimens in retaining the most volatile or gaseous fission products

It is estimated that the specimens reached a temperature of 100-200$^{\circ}$ C during irradiation so that some gaseous products may have been lost at this time A specimen was sealed in glass during exposure and it was found that about 22% of the Xe calculated to have been generated was lost

from a 'standard' powder sample in the 7 day exposure Losses from the ceramic specimens would be dependent on the diffusion rate of I and Xe in these ceramics at this temperature This rate has been calculated to be very low so that losses from specimens other than the loose powder "standards' have been assumed negligible

As shown in Table I, the amounts of fission products collected in these tests from the 'standard" samples are between 46% and 118% of the amounts calculated to have been generated 62% of the generated $Xe^{133}$, for example, was actually measured in the radiochemical analyses These data are corrected to a specific time - in this case, the time of removal of the sample from the furnace This amount of $Xe^{133}$ plus the 22% found lost during exposure accounts for 84% of the calculated amount generated This indicated a reasonably good efficiency in the collecting system

Because of the nature of the test, the very short half life isotopes could not be measured It was not convenient to measure $\beta$ activity for the majority of the samples, although Table II shows the results of washing a collection tube from sample G-33-3, which is representative of the $\beta$ measurements made

The data in Table III show the ratios of the activity collected from the ceramic specimens to the activity collected from the appropriate "standard' The Cr-$Al_2O_3$ specimens (A) were prepared from powders prepared in a tungsten carbide ball mill The resulting tungsten impurity formed a volatile oxide on the surface during heating which contributed nearly all the measurable activity The carbide cermets (B, C) were found to have the uranium distributed uniformly

throughout them which presumably resulted from diffusion during the sintering step of their fabrication Since there was fissionable material on and near the surface, much more activity was noted The (D) specimens, MgO-Ni, showed the very lowest activity at 900° C while the (E) specimens, SiC-Si, were not quite as good It must be pointed out, however, that there was a small amount of U exposed on the surface of the (E) specimens due to imperfect fabrication

Where the activity resulting from diffusion in the ceramics was identified, it was found to be Xe, Te and I Under the conditions of this test, it is estimated that the best ceramic fuel elements will retain most of these and other fission products generated Approximately 0 001% to 0 01% of the volatile fission products will escape

Table IV shows that Np was found on the surface of the specimens, and is roughly proportional to uranium surface contamination Np was also found in the aluminum silicate wool surrounding the specimens

Table V shows representative data obtained from some of the (D) specimens These data are self-explanatory The last column indicates that in general more of the activity is condensed in that portion of the collecting tube which was deepest in the liquid nitrogen and therefore the coldest

Methods of calculation, equations used and references are given in the appendices

Acknowledgments

The work reported here was done as a summer project by W E Moody, Jr It resulted from earlier work by J R Johnson and A J Taylor, who devised the method and made a number of preliminary experiments not included in this report

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TABLE I

TABULATED RESULTS FOR STANDARD COMPACTS

| Compact Specimen No | Isotope | Measured Activity Photons/min | Activity Corrected to d/min (7) | Number of Atoms Diffused thru at time of removal from furnace(6) $N_1 \times 10^{10}$ | Theoretical total Number of Atoms at time of removal from furnace(5)*** $N_o \times 10^{10}$ | % efficiency of collecting system % |
|---|---|---|---|---|---|---|
| G-33-3** | $Xe^{133}$ | $3 \times 10^6$ | $9 \times 10^6$ | 11 0 | 17 72 | 62 |
| | $I^{133}$ | $2 \times 10^6$ | $2\ 2 \times 10^6$ | 1 15 | 1 72 | 68 |
| | $Te^{132}$ | $3 \times 10^6$ | $3 \times 10^6$ | 2 68 | 8 85* | 75 |
| | $I^{132}$ | $7 \times 10^6$ | $7 \times 10^6$ | 2 62 | 2 20 | 118 |
| | $I^{131}$ | $3 \times 10^6$ | $3\ 75 \times 10^6$ | 7 02 | 15 2 | 46 |
| | $Mo^{99}$ | $3 \times 10^6$ | $3\ 3 \times 10^6$ | 2 68 | | |
| | | | Activity Ratio to G-33-3(1) | $UO_2$ Ratio | Efficiency Ratio Change | |
| G-224-1(4) | $Xe^{133}$ | $3 \times 10^7$ | 10 | 6 8 | 1 47 | 91 |
| | $I^{133}$(2) | $2\ 5 \times 10^7$ | 11 5 | | 1 69 | 115 |
| | $Te^{132}$(3) | $1\ 7 \times 10^7$ | 5 7 | | 838 | 63 |
| | $I^{132}$ | $3 \times 10^7$ | 4 3 | | 633 | 75 |
| | $I^{131}$ | $2 \times 10^7$ | 6 7 | | 985 | 45 |
| | $Mo^{99}$ | $1\ 5 \times 10^7$ | 5 0 | | 736 | |

(1) Activity Ratio to G-33-3 x % Efficiency of G-33-3 = Efficiency of G-224-1 $UO_2$ Ratio
(2) Some Ru-103 may contribute
(3) Some Xe-133 m, -135 may contribute $\gamma$ of this energy
(4) Rough calibration, absolute error probably less than 50%
(5) Calculated using data of LRL-153, Appendix 'B'
(6) Sample calculation Appendix 'A"
(7) Conversion Factors given in Appendix "A", Table IV

* Calculated from data and not LRL-153
** Detailed Radiochemical Analysis of 87% of the activity dissolved from tube in dilute nitric acid given in Table II
*** These calculations are dependent on numerous factors which may include experimental errors or uncertainty The exact reactor flux, for instance, is not known

TABLE II

RADIOCHEMICAL ANALYSIS OF

G-33-3 NITRIC ACID WASH FROM TUBE*

| Component | Counts/Min /Ml | % of Gross |
|---|---|---|
| Gross $\gamma$ | $7\ 7 \times 10^4$ | -- |
| Te $\gamma$ | $2\ 9 \times 10^4$ | 38 |
| I $\gamma$ | $2\ 0 \times 10^4$ | 26 |
| Mo $\gamma$ | $6\ 3 \times 10^3$ | 8 |
| Rare earth $\gamma$ | 810 | 1 |
| Ba $\gamma$ | 186 | 0 2 |
| Zr $\gamma$ | 100 | 0 1 |
| Gross $\beta$ | 7130 | -- |
| Rare earth $\beta$ | 113 | 1 6 |
| Sr $\beta$ | 45 | 0 6 |

* Determination made after approximately one week decay from time of removal from furnace or 10 days from time of removal from irradiation

TABLE III

TABULATED RESULTS FOR CERMET COMPACTS

| Compact Specimen No | Temp °C of 24 Hr Heating | Type of Activity Detected as Products of Diffusion | Ratio of Diffused Activity to Activity of G-33-3 Standard (Fission Product) (Retention Factor) | Compact Composition | Uranium Compound in Compact | Remarks |
|---|---|---|---|---|---|---|
| A33-5 | 1000° C | ($W^{187}$ in all A | $2.4 \times 10^{-1}$ | ($Al_2O_3$ -28% | $UO_2$ | $W^{187}$ contamination in |
| A33-7 | 1000° C | (samples (forming | $1.5 \times 10^{-3}$ | (Cr -72% | 3.3 mg | all A specimens due to |
| A33-3 | 900° C | (impurity) and con- | $2 \times 10^{-4}$ | | | grinding media contam- |
| A33-4 | 900° C | (stituted majority | $4 \times 10^{-4}$ | | | ination in specimen |
| | | (of activity Xe | | | | preparation |
| | | (and I present | | | | |
| A33-1 | 800° C | (Not identified | $4 \times 10^{-5}$ | | | |
| A33-2 | 800° C | ( " " | $8 \times 10^{-5}$ | | | |
| B33-2 | 1000° C | (Xe, Te, I | $3.3 \times 10^{-2}$ | (ZrC -80% | $UO_2$ | $UO_2$ distributed |
| B33-5 | 1000° C | ( " " | $1.5 \times 10^{-2}$ | (Fe -20% | 3.3 mg | throughout specimen |
| C33-3 | 1000° C | (Xe, Te, I | $1 \times 10^{-1}$ | (TiC -80% | $UO_2$ | $UO_2$ distributed |
| C33-6 | 1000° C | ( " " " | $6 \times 10^{-2}$ | (Ni -20% | 3.3 mg | throughout specimen |
| D33-7 | 1000° C | (Xe, Te, I* | $2 \times 10^{-3}$ | (MgO -34% | $UO_2$ | |
| D33-9 | 1000° C | ( " " | $3 \times 10^{-3}$ | (Ni -66% | 3.3 mg | |
| D33-1 | 900° C | (Not identified | $4 \times 10^{-5}$ | | | |
| D33-2 | 900° C | ( " " | $9 \times 10^{-5}$ | | | |
| D33-3 | 800° C | Specimen used for Np | | | | |
| D33-4 | 800° C | Not identified | $2 \times 10^{-5}$ | | | |
| E224-1 | 900° C | (Xe, Te, I, trace | $7 \times 10^{-4}$ | SiC bonded | ( $USi_3$ | Stringer of $USi_3$ to |
| E224-2 | 900° C | ($Au^{198}$ in E224-1 | $9 \times 10^{-4}$ | with Si | (26.5 mg | surface of specimen |
| E224-3 | 900° C | No elements could | $2 \times 10^{-4}$ | | ( $UO_2$ | Therefore, surface of |
| | | be identified | | | (22.4 mg | specimen contaminated |

*Complete Analyses given in Data

TABLE IV

Np RESULTS

| Compact Specimen No | Np Activity in Acid Wash Counts/min/ml | Uranium in Acid Wash(3) $\mu$g/ml | Temperature Specimen Subjected to |
|---|---|---|---|
| A-33-3 | 636 | 015 | 900° C |
| A-33-1 | 3 15 x $10^3$ | 057 | 800° C |
| B-33-2 | 1 54 x $10^6$ | 138 0 | 1000° C |
| D-33-7 | 1 53 x $10^4$ | 41 | 1000° C |
| D-33-3(1) | 1 87 x $10^4$ | 1 85 | 150° C |
| D-33-3(2) | 5 4 x $10^3$ | 21 | 800° C |
| E-224-1 | 1 26 x $10^4$ | 092 | 900° C |

(1) After removal from neutron flux
(2) After heating
(3) Fluorimetric analysis

TABLE V

REPRESENTATIVE DATA

| Compact Specimen No | Time[1] Days | Scintillation Counting Data c/min | Temperature of Heating °C | Elapsed time from flux to Furnace Removal Hr | Elapsed time from furnace removal to measurement time Hr | Approx Ratio to G-33-3 | Sealed Tube Top/Bottom Ratio[2] |
|---|---|---|---|---|---|---|---|
| D-33-7 | 1 1 | 10,800 | 1000° | 48 0 | 27 4 | 0019 | |
| | 1 3 | 10,000 | | | | 0019 | |
| | 2 1 | 8,330 | | | | 0019 | |
| | 2 3 | 7 900 | | | | 0019 | |
| | 5 1 | 4,070 | | | | 0018 | |
| D-33-9 | 2 1 | 14,500 | 1000° | 72 0 | 27 4 | 0033 | |
| | 2 3 | 13,500 | | | | 0032 | |
| | 5 1 | 6 800 | | | | 0030 | |
| D-33-1 | 1 | 316 ± 16 | 900° | 25 0 | 47 5 | -- | 0 6 |
| | 25 | 409 ± 16 | | | | -- | 0 7 |
| | 1 0 | 242 ± 15 | | | | $4 \times 10^{-5}$ | 0 6 |
| | 1 3 | 184 ± 15 | | | | $3 \times 10^{-5}$ | 1 3 |
| | 2 0 | 165 ± 15 | | | | $4 \times 10^{-5}$ | 1 1 |
| | 2 3 | 138 ± 15 | | | | $3 \times 10^{-5}$ | 1 2 |
| D-33-2 | 1 2 | 548 ± 16 | 900° | 49 0 | 28 3 | $10 \times 10^{-5}$ | 1 7 |
| | 1 3 | 509 ± 16 | | | | $10 \times 10^{-5}$ | 1 7 |
| | 2 0 | 410 ± 15 | | | | $9 \times 10^{-5}$ | 1 8 |
| | 2 3 | 333 ± 16 | | | | $8 \times 10^{-5}$ | 1 9 |
| D-33-3 | Specimen used for Np | | | | | | |
| D-33-4 | 1 1 | 107 ± 15 | 800° | 50 0 | 24 4 | $1 9 \times 10^{-5}$ | 1 2 ± 3 |
| | 1 3 | 131 ± 14 | | | | $2 4 \times 10^{-5}$ | 1 2 ± 3 |
| | 2 0 | 104 ± 14 | | | | $2 3 \times 10^{-5}$ | 1 2 ± 3 |
| | 2 1 | 73 ± 14 | | | | $1 7 \times 10^{-5}$ | 1 0 ± 4 |
| | 2 3 | 65 ± 14 | | | | $1 5 \times 10^{-5}$ | 0 9 ± 4 |
| | 6 0 | 50 ± 13 | | | | $2 5 \times 10^{-5}$ | 0 7 ± 4 |

(1) Days from 9 30 A M Wednesday reference time

(3) End of rube closest to compact designated as Bottom

# APPENDIX "A"

## APPROXIMATE

## CONVERSION FACTORS

| Isotope | Conversion Factor | | | | |
|---|---|---|---|---|---|
| $Xe^{135}$ | Photons/min | | x 1 | = | d/min |
| $Xe^{133}$ | " | " | x 3 | = | " |
| $I^{133}$ | " | ' | x 1 1 | = | ' |
| $Te^{132}$(4) | ' | " | x 2 5 | = | " |
| $I^{132}$ | " | ' | x 1 | = | ' |
| $I^{131}$ | " | " | x 1 25 | = | " |
| $Mo^{99}$ | " | " | x 1 1 | " | ' |

1 mv on Ion Chamber = 1 37 $\pm$ 03 x $10^4$ c/min on Scintillation Counter

# APPENDIX "A'

## CALCULATION OF $Xe^{133}$

The amount of $Xe^{133}$ which diffused through and became trapped at the time of removal from the furnace was calculated from standard radioactivity equations As $Xe^{133}$ was in the same decay chain as $I^{133}$, it was necessary to make calculations for both isotopes

1 Amount present at time of measurement

$$A = -\lambda N$$

$$N = \frac{A}{\lambda}$$

A = activity

$\lambda$ = declay constant

N = number of atoms

$$N_{Xe} = \frac{A}{\lambda} = \frac{9 \times 10^{6}}{9\ 09 \times 10^{-5}} = 9\ 9 \times 10^{10}$$

$$N_{I} = \frac{A}{\lambda} = \frac{2\ 2 \times 10^{6}}{5\ 29 \times 10^{-4}} = 417 \times 10^{10}$$

2 Amount present at time of removal from furnace

Elapsed time for furnace to measurement = 32 3 hr

I is the parent of Xe

$$N = N_{o}e^{-\lambda t}$$

$$N_{I_o} = \frac{417 \times 10^{10}}{e^{-(3\ 18 \times 10^{-2}\ hr^{-1})(32\ 3\ hr)}} = 1\ 15 \times 10^{10}$$ atoms of $I^{133}$ at time of removal from furnace

## APPENDIX A'

The decay equation for parent and daughter is

$$N_2 = \frac{\lambda_1}{\lambda_2 - \lambda_1} N_{1_o} (e^{-\lambda_1 t} - e^{-\lambda_2 t}) + N_{2_o} e^{-\lambda_2 t}$$

$N_2$ = number of atoms of daughters at time t

$\lambda_2 = 5\ 46 \times 10^{-3}\ hr^{-1}$

$$9\ 9 \times 10^{10} = \frac{\lambda_1}{\lambda_2 - \lambda_1} (1\ 15 \times 10^{10})(e^{-\lambda_1 t} - e^{-\lambda_2 t}) + N_2 o_e^{-\lambda_2 t}$$

$$9\ 9 \times 10^{10} = (1\ 21)(1\ 15 \times 10^{10})\ (\ 479) = 839\ N_2 o$$

$N_2 o = 11\ 00 \times 10^{10}$ atoms $Xe^{133}$ at time of removal from furnace for amount diffused through specimen G-33-3

## APPENDIX "B"

Fission of Natural Uranium

The specimen contained 3 3 mg of $UO_2$

Natural uranium contains 0 719% of $U^{235}$

$$\frac{\text{U mole wt}}{UO_2 \text{ mole wt}} \times \text{amount } UO_2 \times \% \ U^{235} = \text{amount } U^{235}$$

$$\frac{238}{270} \times 0033 \text{ gm} \times 00719 = 20\ 92 \times 10^{-6} \text{ gm } U^{235}$$

$$= 5\ 37 \times 10^{16} \text{ atoms } U^{235}$$

$$= 1\ 152 \times 10^{20} \text{ atoms } U^{235}/cm^3$$

$$\text{Volume} = \frac{0033}{7\ 31} = 4\ 65 \times 10^{-4} \ cm^3$$

$$\text{Reaction Rate} = \sigma_f \ N_o \phi$$

$$\phi = 5 \times 10^{11} \ n/cm^2 \ sec$$

$$\sigma_f = 574 \times 10^{-24} \ cm^2$$

$$N_o = 1\ 152 \times 10^{20} \ atom/cm^3$$

$$\text{Reaction Rate} = (5\ 74 \times 10^{-24} \ cm^2)(1\ 152 \times 10^{20} \ atom/cm^3)(5 \times 10^{11} \ n/c^2 \ sec)$$

$$= 33\ 1 \times 10^9 \ f/cm^3 \ sec = (33\ 1 \times 10^9)(4\ 65 \times 10^{-4}) \ f/sec$$

$$= 15\ 4 \times 10^6 \ f/sec$$

[redacted]



## APPENDIX 'B

### CALCULATION OF $Xe^{133}$ FORMED AND PRESENT AT TIME OF REMOVAL FROM FURNACE USING DATA OF LRL-153(2)

1 Power Factor

The data of LRL-153 was based on a 2000 watt reactor The power for the specimen was much lower and a correction factor was introduced

$3\ 1 \times 10^{10}$ f/sec = 1 watt

$\frac{\text{f/sec for specimen}}{\text{conversion factor}}$ = 1 power of specimen

$\frac{15\ 4 \times 10^{6}\ \text{f/sec}}{3\ 1 \times 10^{10}\ \text{f/sec watt}} = 5 \times 10^{-4}$ watts

$\frac{5 \times 10^{-4}\ \text{watts}}{2000\ \text{watts}} = 2\ 5 \times 10^{-7}$ correction factor

2 Activity of $Xe^{133}$

Build up time = 1 week = $1\ 008 \times 10^{4}$ min

Decay time = 97 hr = $5\ 82 \times 10^{3}$ min

From the LRL-153 data, 2 90 curies of $Xe^{133}$ was formed

Report amount present x Power factor correction = amount present

2 90 curies x $2\ 5 \times 10^{-7} = 7\ 25 \times 10^{-6}$ curies

$A = \lambda N$ $\qquad N = \frac{A}{\lambda}$

$$N = \frac{(7\ 25 \times 10^{-6}\ \text{curies})(3\ 7 \times 10^{10}\ \text{c/sec curies})}{1\ 515 \times 10^{-6}\ \text{sec}^{-1}}$$

= $17\ 72 \times 10^{10}$ atoms of $Xe^{133}$ present at time of removal from furnace

[redacted]

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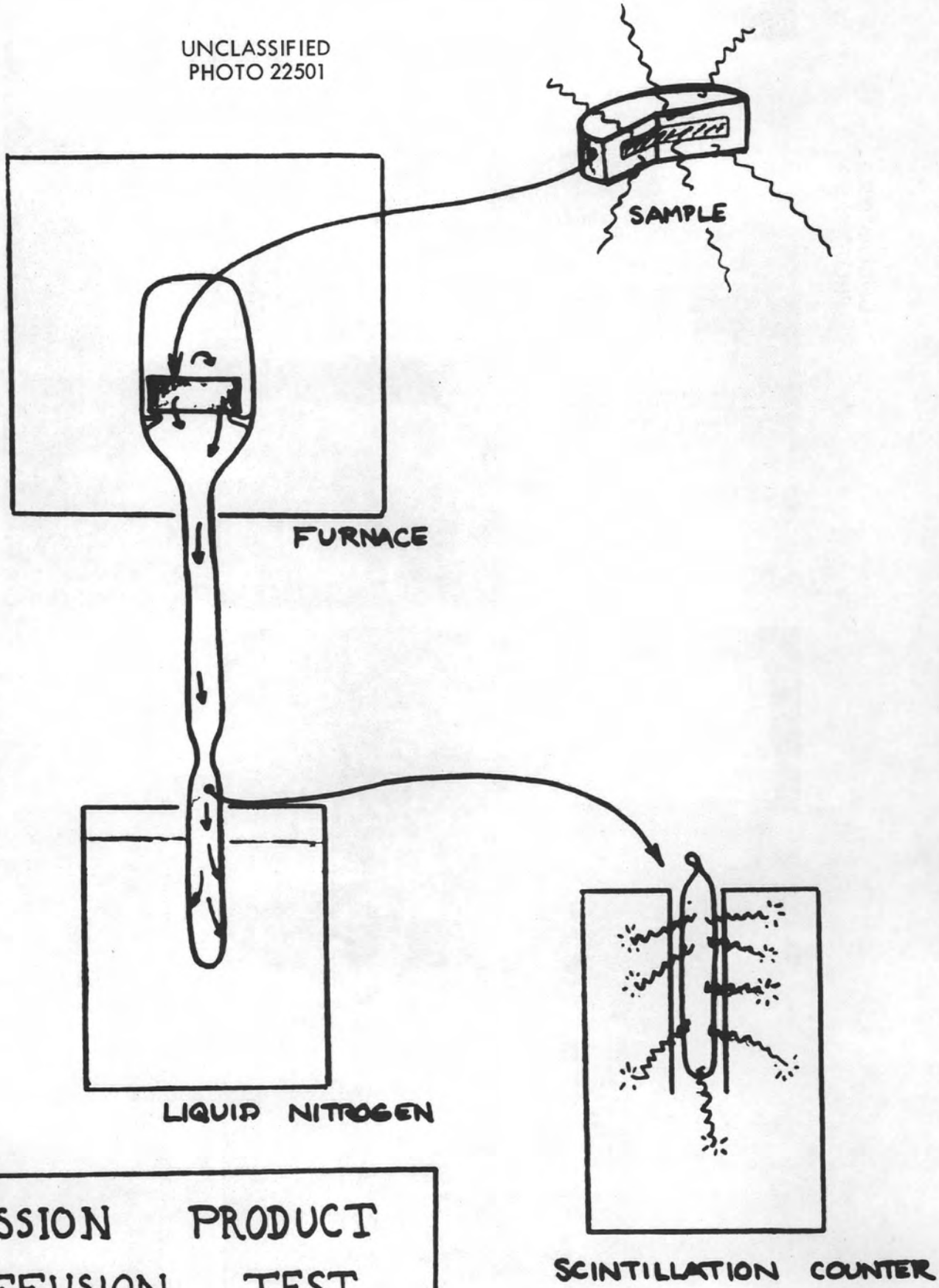


FISSION PRODUCT DIFFUSION TEST

FIGURE 1

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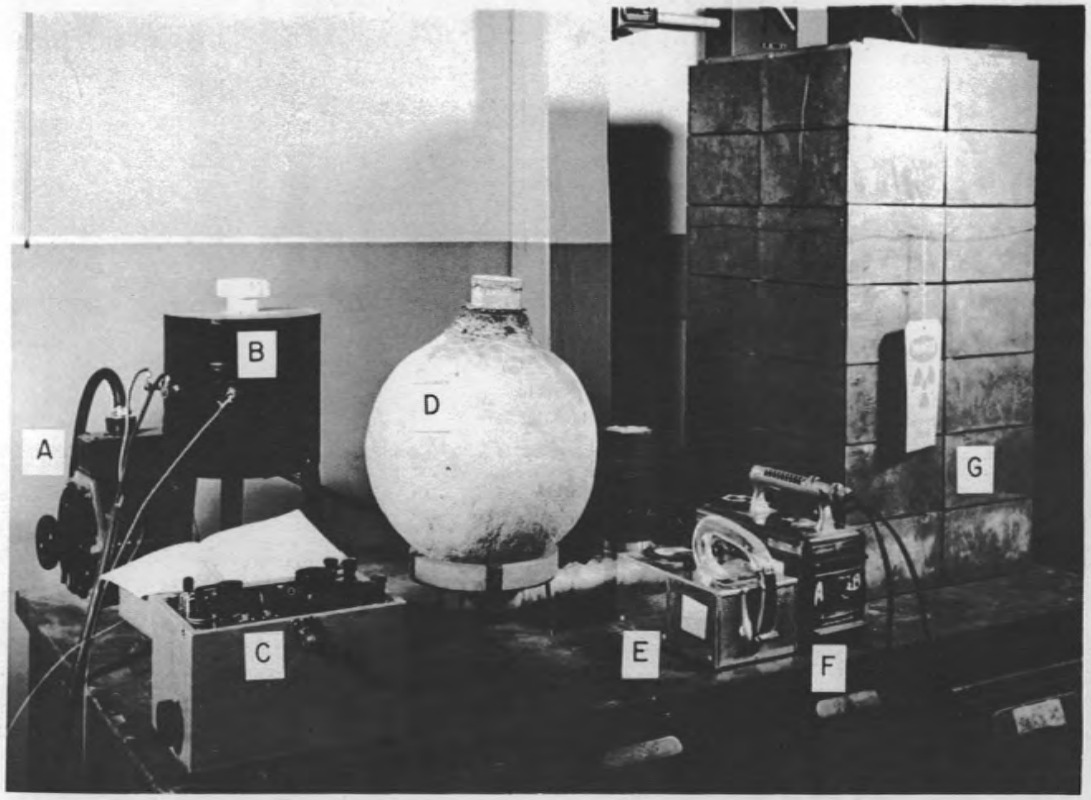


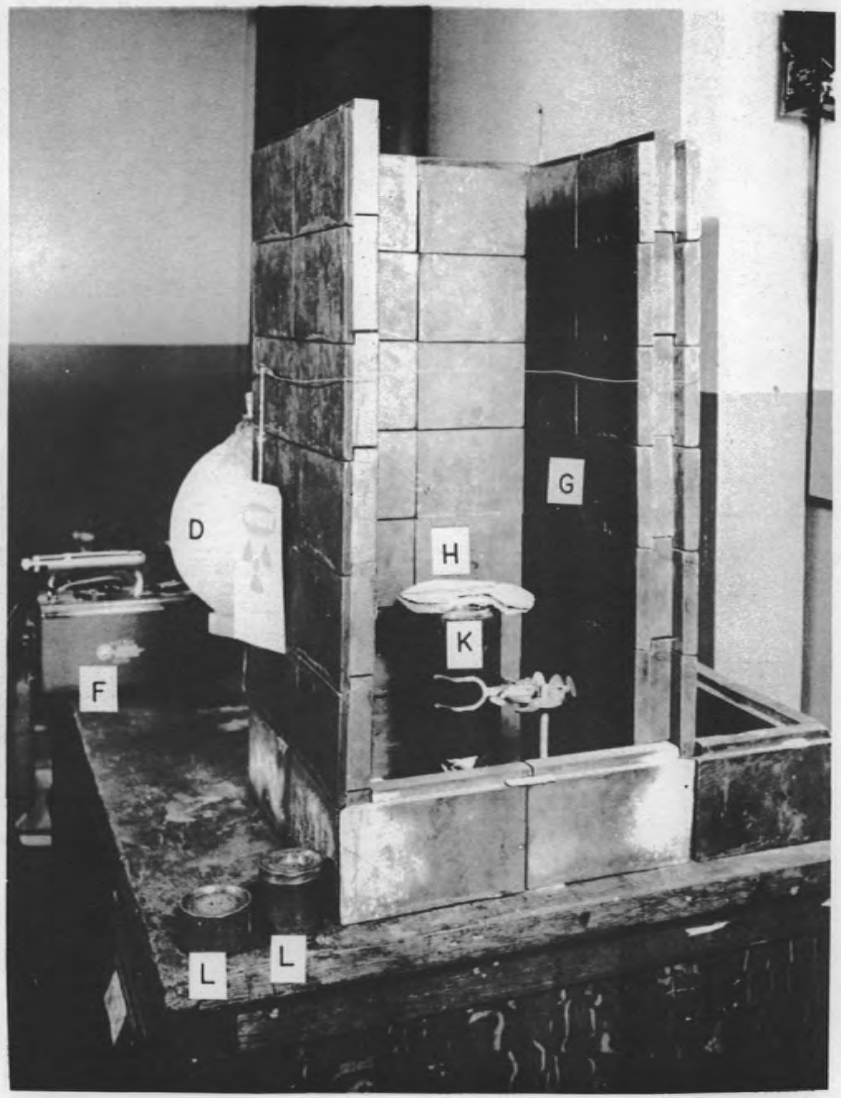


A-Variac , B-Wire-wound Furnace, C-Potentometer, D-Dewar Flask
E-Counter (Alpha, Beta, Gamma), F-Counter (Beta, Gamma), G-Lead Brick Housing, H-Insulating Disc, K-Dewar Flask, L-Lead Containers.

FIGURE 2. APPRATUS AND EQUIPMENT

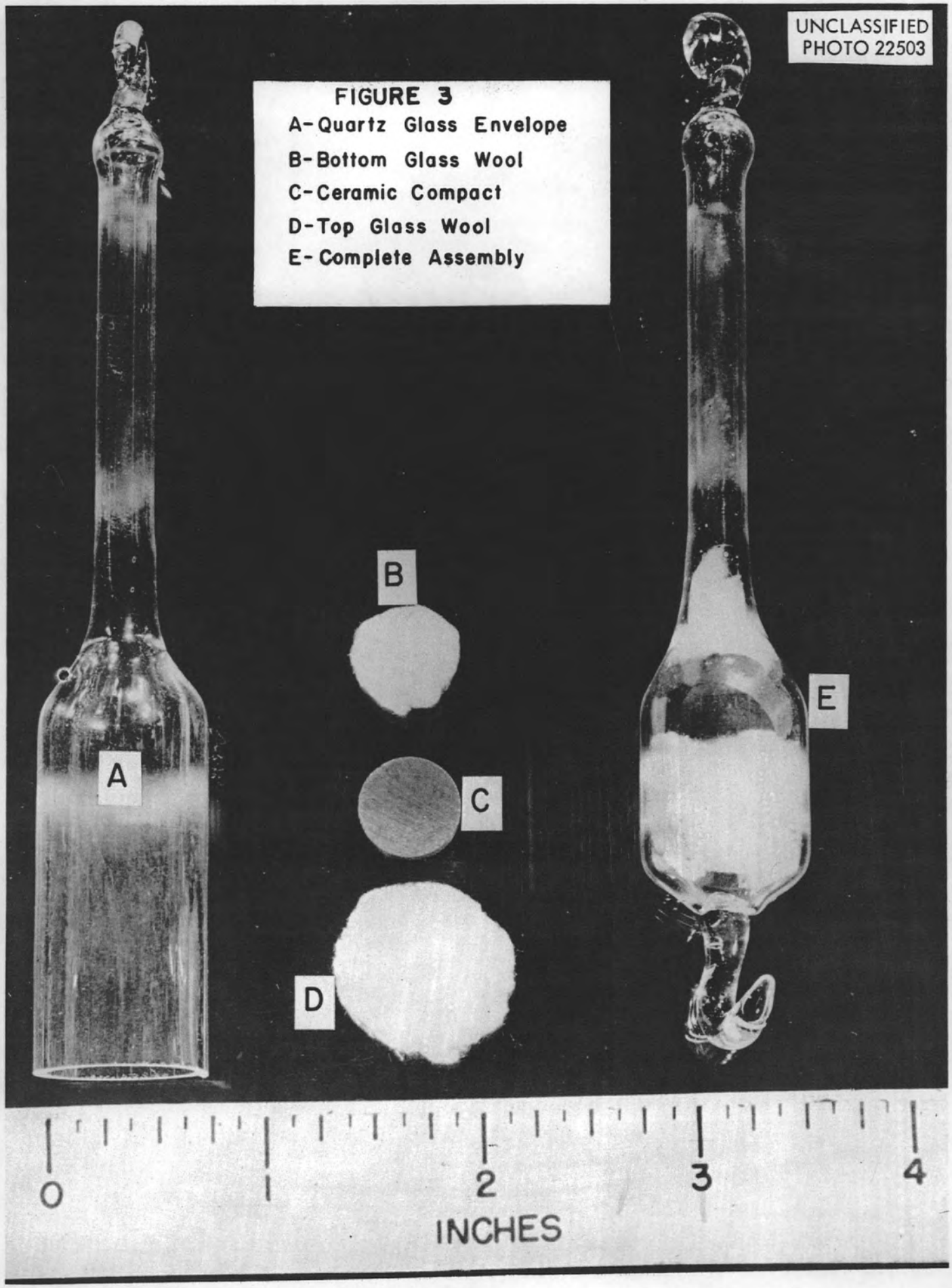


FIGURE 3
A-Quartz Glass Envelope
B-Bottom Glass Wool
C-Ceramic Compact
D-Top Glass Wool
E-Complete Assembly

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PHOTO 22504

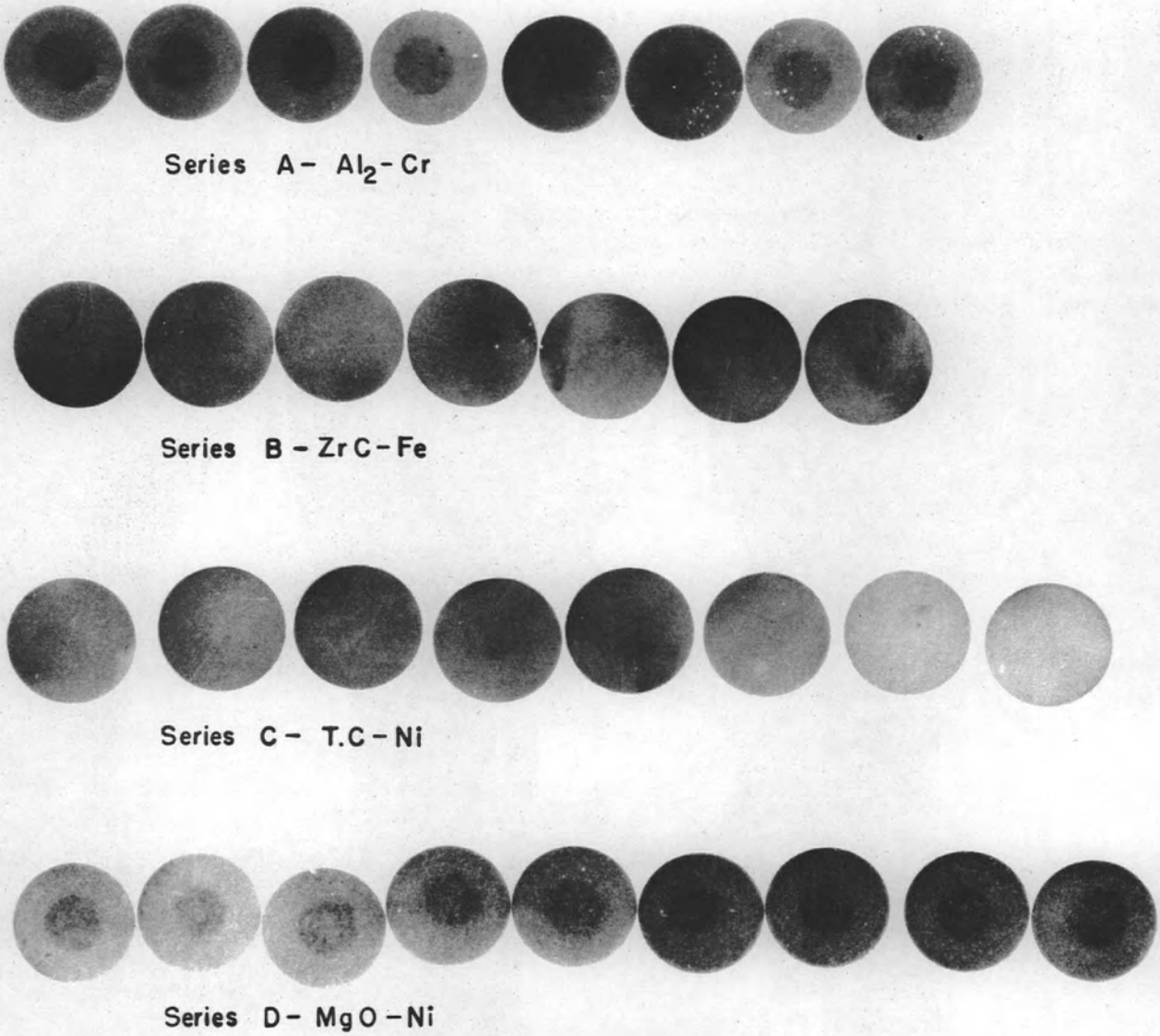


FIGURE 4. RADIOGRAPHS OF CERMETS