THE FABRICATION AND EVALUATION OF SAMPLES FOR THE
IRRADIATION PORTION OF THE FBR PHYSICS CONSTANTS PROGRAM

by

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ABSTRACT

The contents of both irradiation samples and calibration standards were evaluated by means of measurements of reactivity, neutron activation, and by neutron radiography. The contents of the irradiation samples were found to be as quoted by the manufacturer. The pure absorber samples (lead, aluminum, copper, niobium, and gold) were found to be satisfactory except for the 100% gold sample which was heavily self-shielded. The UO_2 -Al samples were found to be unsatisfactory because of uncertainty as to their contents.

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I. INTRODUCTION

The irradiation portion of the FBR Physics Constants Program at the National Reactor Testing Station is being undertaken by Aerojet Nuclear Company for the purpose of evaluating the reactivity of mixed oxide fuels as a function of burn-up. The gross reactivity response is a complicated function of fuel burn-up, fuel production, fission product build-up and decay, neutron spectrum, and sample density. It is not enough, therefore, to simply measure the reactivity response before and after an irradiation. One would like to know the flux spectrum as well as level during irradiation and certainly the flux spectrum in which the reactivity measurement is made. Some non-destructive method of analysis for fuel and absorber content is also needed. To solve these problems a variety of techniques have been used by other programs and are well developed. Reactivity measurements have been made in two high-precision instruments: the Advanced Reactivity Measurement Facility^[1] (ARMF) and the Coupled Fast-Reactivity Measurement Facility^[2] (CFRMF). The flux spectra in the measuring reactors has been characterized by calculational, proton-recoil, and foil activation methods; and the irradiation flux level and spectrum have been measured by foil activation. Non-destructive analyses for content have been made by a multi-location method of measurement^[3] in the ARMF I. Gross reactivity has been measured in the CFRMF.

The samples needed for this work consist of two types: the irradiation samples and reactivity calibration standards. Dimensionally, these two types are identical. They differ only in the nature of the active material they contain. This report, then will describe the

various types and the methods that were used to assure that each individual sample did indeed contain the materials, in the amounts specified.

II. FABRICATION

The sample capsule is shown pictorially in Figure 1. This design was dictated by the following considerations:

1. The capsule must fit in a B-19 fuel pin supplied by the EBR-II project of Argonne National Laboratory (ANL).
2. The assembled experiment must pose no hazard to the safe operation of the EBR-II reactor.
3. The reactivity worth of the individual samples must be such that they can be safely measured in both the ARMF-I and the CFRMF.
4. They must be individually identifiable by marking that would be permanent yet would not weaken the sample enclosure.
5. They must not exceed safe operating temperatures during irradiation.

A final requirement was imposed by the desire to spend as little time as possible on sample procurement. This last requirement meant that if an already designed sample could be found, much time would be saved. Fortunately, a supply of 20% PuO_2 - UO_2 mixed oxide fuel pellets was found and the sample capsule was designed around these pellets.

As it turned out item 3 above proved to be the most difficult requirement. The reason is that the reactivity worths in $\Delta k/k$ per gram of pellet are vastly different in the ARMF I and the CFRMF. A sample large enough to give a significant response in the CFRMF would be much too large for safety in the ARMF I. It so happened, however, that this supposed difficulty was a blessing in disguise. The axial distribution of flux in the EBR II is not flat. Therefore, different parts of a

FIGURE 1. Sample capsule for the irradiation portion of the FBR Physics Constants program.

FIGURE 2. Assembly view of sample capsules loaded in a B-19 type EBR-II irradiation capsule.

single long sample would see different flux levels and possibly different spectra during irradiation. Thus, by encapsulating smaller numbers of pellets and stacking them in the B-19 fuel pin a range of Nvt can be obtained during a single irradiation period. Each of these smaller samples are, therefore, measurable in the ARMF I for sample content analysis and flux-foils can be located at intervals along the sample string during irradiation. These same samples can then be assembled in a container for measurement of gross reactivity in the CFRMF.

Figure 2 shows the assembled sample string including empty and loaded capsules.

Requirements 1 and 2 are described in detail in the users Guide for Irradiation Experiments in EBR-II. Requirement 4 is met by etching the small tab shown in Figure 1. Calculations submitted as required by items 1 and 2 have shown that sample heating during irradiation does not exceed acceptable levels.

The mixed oxide fuel pellet specifications are given in Table I. Four of these were loaded into a capsule in a helium atmosphere and sealed by heliarc welding. Each capsule was then x-rayed, dye-penetrant leak-tested, neutron radiographed, gamma counted, and the reactivity measured in the ARMF I. The results are discussed in the section on evaluation.

Reactivity calibration samples were fabricated by essentially the same process as the irradiation samples except that the active contents were made by compacting pure and diluted powders of various absorber materials. Table II lists the active materials used in these samples. The completed samples were leak-tested, and the reactivities measured.

The mixtures were also analyzed by activation to confirm the specified content.

III. EVALUATION

The success of this project is critically dependent on a knowledge of the content of the samples to be irradiated. Therefore, several independent methods were used to measure both relative and absolute ^{239}Pu content. The content of the calibration standards is equally important, but since these are non-radioactive, or only slightly so, their content is easy to measure by simple weight.

A. Irradiation Samples

One of the methods for measuring the ^{239}Pu content is to count the gamma rays produced by the natural decay of this isotope. The 413.7 keV photopeak having a branch ratio of $0.0015 \pm 0.0001\%$ was counted and used in the equation,

$$Cs = \lambda N_{\text{Pu}} \beta e \quad (1)$$

where Cs = counts per second

λ = decay constant

N_{Pu} = number of atoms of ^{239}Pu

β = branch ratio of 413.7 keV gamma ray

e = efficiency including self-absorption losses in the sample and sample cladding.

The ^{239}Pu content of the samples can be obtained from these counting data by use of the equation,

$$W_{\text{Pu}} = N_{\text{Pu}} A_{\text{Pu}} / N_{\text{O}} \quad (2)$$

where A_{Pu} = the atomic weight = 239.0522

N_{O} = Avogadro's number = 6.02253×10^{23}

The values of the constants used were

$$\lambda = 9.01663 \times 10^{-13} \text{sec}^{-1} \quad (\tau_{1/2} = 24,360 \text{ yr})$$

$$\beta = 1.5 \pm 0.1 \times 10^{-5}$$

$$e = 0.74946 \times 10^{-4} \quad (\text{see Appendix I})$$

When these values are substituted into eqns 1 and 2 we get

$$W_{\text{Pu}} = C_s \times 0.391588 \quad (3)$$

The results are shown in Table III.

TABLE I a

Specification for Mixed Oxide Fuel Pellets

Dimensions: 0.213" $\pm$ 0.001" diameter
0.250" $\pm$ 0.002" long
1.5 $\pm$ 0.01g weight
93.1 $\pm$ 1.0% theoretical density

Composition: 20 w/o PuO₂, 80 w/o UO₂ (nominal)
Uranium - depleted
Plutonium - enriched

Analysis: Pu - 17.8 w/o
U - 70.3 w/o
O - 11.8 w/o
Total impurities < .1%

Isotopic:

Pu-238	0.11%
239	88.27
240	9.41
241	2.02
242	0.19
U-234	< 0.01
235	0.41
236	< 0.01
238	99.58

Table I b

<u>Sample No.</u>	<u>Fuel Sample Weights</u> <u>Capsule Wt.</u>	<u>Content Wt.</u>
002	10.4528 g	6.0021 g
004	10.4679	6.0049
007	10.4677	5.9997
009	10.4828	5.9989
010	10.4759	5.9971
013	10.4521	5.9999

TABLE II
Descriptions of Calibration Standards

Empty Capsules:

Material: 304 SST

<u>Capsule Number</u>	<u>Weight</u>	<u>Volume (displacement)</u>
003	10.556 g	1.994 cc
004	10.352	1.981
008	10.549	1.988

Absorber Samples:

<u>Sample No.</u>	<u>Capsule Wt.</u>	<u>Sample Mat'l</u>	<u>Mat'l Wt.</u>
018	10.511 g	Lead	4.957 g
019	10.492	Copper	4.661
020	10.424	Aluminum	1.408
021	10.461	Niobium	3.946
023	10.381	4.886 w/o Au	0.078
		95.113 w/o Al	1.520
024	10.527	10.106 w/o Au	0.167
		89.894 w/ Al	1.482
025	10.495	Gold	9.855
027	10.238	5.04 w/o UO ₂	0.079
		94.96 w/o Al	1.486
028	10.327	5.04 w/o UO ₂	0.081
		94.96 w/o Al	1.523
029	10.278	10.00 w/o UO ₂	0.163
		90.00 w/o Al	1.470
030	10.292	10.00 w/o UO ₂	0.164
		90.00 w/o Al	1.476
031	10.220	14.99 w/o UO ₂	0.251
		85.01 w/o Al	1.421
032	10.232	14.99 w/o UO ₂	0.258
		85.01 w/o Al	1.463
033	10.240	20.00 w/o UO ₂	0.351
		80.00 w/o Al	1.404
034	10.294	20.00 w/o UO ₂	0.353
		80.00 w/o Al	1.413
035	10.335	25.00 w/o UO ₂	0.475
		75.00 w/o Al	1.424
036	10.276	25.00 w/o UO ₂	0.447
		75.00 w/o Al	1.342

TABLE III

Measured Sample Contents

<u>Sample No.</u>	<u>Counts/sec</u>	<u>Weight ^{239}Pu (meas.)</u>	<u>Weight ^{239}Pu (Mfg. value)</u>
002	2.4544	0.96 ± 0.06 g	0.9425
004	2.4069	0.94 ± 0.06	0.9430
007	2.4138	0.95 ± 0.06	0.9421
009	2.4577	0.90 ± 0.06	0.0420
010	2.4485	0.90 ± 0.06	0.9422
013	2.4721	0.91 ± 0.06	0.9422

Reactivity measurements on these samples provide only relative information as to their content. The results are shown in Table IV.

TABLE IV

Reactivity Worths of Irradiation SamplesReactor Location in Which Measurement was Made (Fig.3)

<u>Sample No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
002	162.67 μk	298.63 μk	288.76 μk	238.89 μk	37.95 μk
004	162.36	298.66	288.91	238.73	38.43
007	162.06	298.59	288.77	238.75	38.14
009	162.37	299.22	288.76	238.95	38.40
010	162.06	298.03	288.97	238.18	38.24
013	<u>162.53</u>	<u>298.08</u>	<u>288.20</u>	<u>238.37</u>	<u>38.04</u>
Averages	162.34 μk	298.70 μk	288.73 μk	238.81 μk	38.18 μk
Std. dev.	± 0.22	± 0.36	± 0.25	± 0.25	± 0.15
% dev.	0.1%	0.1%	0.09%	0.1%	0.4%

$\mu\text{k} = \Delta k/k \times 10^{-6}$. Standard deviation of repeat measurements on the same sample is about 0.15 μk .

The values in Table IV are total sample reactivities which include capsule plus the active contents. The stainless capsules were very uniform in both weight and water volume displaced as may be seen from the values in Table I-b and II.

A neutron radiograph of the irradiation samples was made at the TREAT facility. The optical density of the resulting film was measured at the center of the image of each of the pellets. Measurements were

TABLE V

Photodensities from Neutron Radiographs(Relative Values)

<u>Sample No.</u>	<u>Density</u>	<u>Background</u>	<u>Difference</u>
002	2.22	3.62	- 1.40
	2.24	3.60	- 1.36
	2.19	3.60	- 1.41
	2.18	3.59	- 1.41
		Average	- 1.405 $\pm$ 0.005
004	2.31	3.76	- 1.45
	2.31	3.76	- 1.45
	2.31	3.76	- 1.45
	2.32	3.77	- 1.45
		Average	- 1.45 $\pm$ 0.00
007	2.31	3.75	- 1.44
	2.32	3.76	- 1.44
	2.33	3.78	- 1.45
	2.32	3.78	- 1.46
		Average	- 1.448 $\pm$ 0.0008
009	2.19	3.62	- 1.43
	2.25	3.67	- 1.42
	2.26	3.69	- 1.43
	2.27	3.70	- 1.43
		Average	- 1.428 $\pm$ 0.0004
010	2.30	3.72	- 1.42
	2.30	3.70	- 1.40
	2.28	3.69	- 1.41
	2.28	3.70	- 1.39
		Average	- 1.405 $\pm$ 0.011
013	2.33	3.79	- 1.46
	2.33	3.76	- 1.43
	2.31	3.75	- 1.44
	2.31	3.75	- 1.44
		Average	- 1.443 $\pm$ 0.011

Average of 24 pellets 1.43 $\pm$ 0.02 (deviation = 1.4%)

also made outside the sample along a line parallel to the axis of the sample. Relative density values are given in Table V.

During the exposure of the neutron radiographic film the samples were loaded in the holder as follows (top to bottom) 009, 007, 004, 013, 010, 002. When listed in this order the average differences are -1.43, -1.45, -1.45, -1.44, -1.41, -1.41. It appears, therefore, that subtraction of the background has not completely removed the bias due to an intensity variation in the irradiating neutron beam. One may conclude from these data that the sample content is constant within the accuracy of the data, i.e., $\approx 1.4\%$

The three methods of evaluating the irradiation samples, reactivity, neutron radiograph, and natural radioactivity indicate therefore that the six samples are uniform as to dimensions and active contents to better than 1%. The discrepancy between measured ^{239}Pu content and that given by the manufacturer as shown in Table III is disappointing, but accountable, since the error limits on the measured value is due entirely to the uncertainty quoted on the value of β . At least the two sets of values are within the uncertainties.

B. Calibration Samples

Samples 018, 019, 020, 021, and 025 contain pure non-radioactive materials and the contents were obtained by direct weighing which is accurate to ± 0.005 g. Of these only the copper, gold and niobium samples have sufficient absorption cross section to result in a significant reactivity response. In the case of gold the pure sample is of little value because of the large sample self-shielding. Further work on dilute gold samples will therefore be needed.

To confirm the expected content of the diluted UO_2 samples a series of activation measurements were made on sample numbers 027 through 033. The ^{239}Np activity was counted as a measure of the ^{238}U content. These measurements proved to be of value only to the extent that the depleted uranium content was shown to differ considerably from the presumed content. These samples are, therefore, of no value to the program. The important point to be noted here is that careful sample preparation does not always yield the desired results and independent evaluation methods are necessary for the confirmation of sample content. Table VI gives the results of the measurements on the non-radioactive samples.

TABLE VI

Reactivity Worths of Absorber Samples

Sample No. 018

Content Mat'l Lead

Content Wt. 4.957 g.

<u>Location (ARMF I)</u>	<u>Net Reactivity^(a)</u>	<u>Capsule Correction^(b)</u>	<u>Sample Net</u>
1	- 53.16 μ k	- 52.93 μ k	- 1.23 μ k
2	- 22.81	- 22.91	+ 0.10
3	- 12.22	- 11.88	- 0.34
4	- 39.43	- 39.67	+ 0.24
5	- 50.42	- 50.55	+ 0.13

Sample No. 019

Content Mat'l Copper

Content Wt. 4.6613 g.

<u>Location (ARMF I)</u>	<u>Net Reactivity</u>	<u>Capsule Correction</u>	<u>Sample Net</u>
1	- 79.04 μ k	- 52.81 μ k	- 26.23 μ k
2	- 35.61	- 22.85	- 12.76
3	- 19.47	- 11.84	- 7.63
4	- 59.25	- 39.58	- 19.67
5	- 70.95	- 50.45	- 20.50

Sample No. 020

Content Mat'l Aluminum

Content Wt. 1.4079 g.

<u>Location (ARMF I)</u>	<u>Net Reactivity</u>	<u>Capsule Correction</u>	<u>Sample Net</u>
1	- 53.60 μ k	- 52.35 μ k	- 1.25 μ k
2	- 23.43	- 22.63	- 0.80
3	- 12.10	- 11.71	- 0.39
4	- 39.43	- 39.23	- 0.20
5	- 51.00	- 50.10	- 0.10

Sample No. 021

Content Mat'l Niobium

Content Wt. 3.9463 g.

<u>Location (ARMF I)</u>	<u>Net Reactivity</u>	<u>Capsule Correction</u>	<u>Sample Net</u>
1	- 58.17 μ k	- 52.60 μ k	- 5.57 μ k
2	- 25.76	- 22.75	- 3.01
3	- 13.34	- 11.78	- 1.56
3	- 43.11	- 39.42	- 3.69
5	- 54.98	- 50.29	- 4.69

Sample No. 023

Content Mat'l

Content Wt. 0.0781 g Au, 1.5187 g Al. (4.887 w/o Au)

<u>Location</u>	<u>Net Reactivity</u>	<u>Capsule Correction</u>	<u>Sample Net</u>	<u>Au Worth</u>
1	- 58.48 μ k	- 52.06 μ k	- 6.42 μ k	- 64.92 μ k/g
2	- 25.47	- 22.49	- 2.98	- 27.02
3	- 13.32	- 11.63	- 1.69	- 16.26
4	- 43.81	- 39.04	- 4.79	- 58.51
5	- 55.50	- 49.88	- 5.62	- 56.72

Sample No. 024

Content Mat'l Gold and Aluminum

Content Wt. 0.1667 g Au, 1.483 g Al (10.106 w/o)

<u>Location</u>	<u>Net Reactivity</u>	<u>Capsule Correction</u>	<u>Sample Net</u>	<u>Au Worth</u>
1	- 65.22 μ k	- 53.04 μ k	- 12.18 μ k	- 65.15 μ k/g
2	- 28.27	- 22.97	- 5.26	- 26.52
3	- 15.36	- 11.91	- 3.45	- 18.24
4	- 48.59	- 39.75	- 8.84	- 51.77
5	- 61.13	- 50.63	- 10.50	- 56.03

Sample No. 025

Content Mat'l Gold

Content Wt. 9.8545

<u>Location</u>	<u>Net Reactivity</u>	<u>Capsule Correction</u>	<u>Sample Net</u>	<u>Au Worth</u>
1	- 231.13 μ k	- 52.83 μ k	- 178.30 μ k	- 18.09 μ k/g
2	- 103.88	- 22.86	- 81.02	- 8.22
3	- 58.29	- 11.85	- 46.44	- 4.71
4	- 171.12	- 39.59	- 131.53	- 13.35
5	- 207.46	- 50.47	- 156.99	- 15.93

- (a) Net reactivity includes corrections for reactor drift and for the worth of the DELRIN capsule holder.
- (b) Capsule correction accounts for the worth of an empty stainless steel capsule. The effect of displacing water is accounted for by this correction. The factors used were obtained from measurements on a series of empty capsules and were found to be as follows:

Loc. 1	$v_i = 8.91 \pm 0.30 \mu\text{k/cc}$	$C_i = 6.722 \pm 0.045 \mu\text{k/cc}$
2	$v_i = 5.75 \pm 0.34$	$C_i = - 3.268 \pm 0.052$
3	$v_i = 4.27 \pm 0.34$	$C_i = - 1.938 \pm 0.049$
4	$v_i = 6.61 \pm 0.40$	$C_i = - 5.025 \pm 0.071$
5	$v_i = 2.08 \pm 0.33$	$C_i = - 5.203 \pm 0.048$

v_i = worth of displaced water

C_i = worth of stainless

IV. CONCLUSIONS

The results of these measurements indicate that the irradiation samples are indeed as uniform in content as reported by the manufacturer, and the activation results indicate that, within the uncertainty of the measurements, the contents are as quoted by the manufacturer. In the case of the pure absorbers there is little reason to question the reported weights. In the case of the two Au-Al samples the results are inconsistent in the various measuring locations. If self-shielding were present one would expect the larger concentration sample to show a smaller reactivity per gram as is evident in the 100% Au samples. As it is, one location⁽³⁾ shows a significantly larger Au-worth in the 10% as compared to the 5% sample; two (2 and 4) show significantly smaller Au-worths; and two (1 and 3) show marginal differences, one being larger and one being smaller in the 10% as compared to the 5% sample. Such inconsistencies are probably due to uncertainties in the capsule correction which has not yet been exhaustively evaluated; or possibly, to inhomogeneities in the sample.

Activation and reactivity measurements on the UO_2 -Al mixtures indicate that they are unsuitable for use in this program.

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APPENDIX

Efficiency factors quoted in the literature on gamma-ray spectroscopy apply to thin sources, i.e., sources which do not themselves absorb gamma rays. The mixed uranium and plutonium oxides used in the irradiation samples, however, absorb a significant quantity of gamma rays. The problem is further complicated by the geometry. Since these samples constitute a source extended throughout a non-zero volume an additional correction must be made.

In the derivation to follow it is assumed that the gamma rays that reach the crystal all arrive in a parallel beam; thus only that part of the total gamma rays emitted in the direction of the crystal are considered. The geometry of the source is illustrated in the sketch. The crystal is assumed to be at a considerable distance below the figure. Figure A-1 is a sectional view of the sample; A-2 is an element of the area shown in A-1.

ρ = the radiation from the area $dy \, dx$
emitted in the downward direction.

μ = attenuation coefficient, cm^2/g

Then, the radiation emitted from the element dx is

$$dA_y = \int_0^L \rho \exp(-\mu y) dy = \frac{\rho}{\mu} [1 - \exp(-\mu L)]$$

and the total activity emitted in the downward direction is

$$A = \int_{-r}^{+r} dA_y \, dx = \frac{\rho}{\mu} \int_{-r}^{+r} [1 - \exp(-\mu L)] dx$$

But $L = 2(r^2 - x^2)^{1/2}$, which when substituted in the above yields

$$A = \frac{\rho}{\mu} \left\{ 2r - \int_{-r}^{+r} \exp[-2\mu(r^2 - x^2)^{1/2}] dx \right\}$$

To evaluate the integral, let $t = (r^2 - x^2)^{1/2}$. Then

$$x = (r^2 - t^2)^{1/2}, \quad dx = -tdt/(r^2 - t^2)^{1/2}$$

$$t = 0, \quad x = \pm r;$$

$$t = \pm r, \quad x = 0$$

When t is substituted in the integral, the result is

$$\begin{aligned} \int_{-r}^{+r} \exp[-2\mu(r^2 - x^2)^{1/2}] dx &= 2 \int_0^{+r} \exp[-2\mu(r^2 - x^2)^{1/2}] dx \\ &= 2 \int_0^r t \exp(-2\mu t) dt / (r^2 - t^2)^{1/2} \end{aligned}$$

This integral in t is given in Gradstein and Ryzhik⁽⁴⁾, formula 3.365 (1) p. 316.

$$\int_0^r t \exp(-2\mu t) dt / (r^2 - t^2)^{1/2} = \frac{\pi r}{r} [L_1(2\mu r) - I_1(2\mu r)] + r$$

The functions $L_1(2\mu r)$ and $I_1(2\mu r)$ are also given in Gradstein and Ryzhik as follows:

$$I_1(z) = \sum_{k=0}^{\infty} (z/2)^{2k+1} / k!(k+1)! \quad z = 2\mu r$$

8.447(2) p. 961

$$L_1(z) = \sum_{m=0}^{\infty} (z/2)^{2(m+1)} / \Gamma(m+3/2)\Gamma(m+5/2), \quad z = 2\mu r$$

8.550 (2) p. 982

The total emission from the section shown in the figure in the absence of absorption is

$$\pi r^2 p = A_0$$

Hence, the correction factor, $1/c = A/A_0$ is

$$1/c = \frac{1}{\pi r^2 \mu} \left\{ 2r - 2 \left(\frac{\pi r}{2} [L_1(2\mu r) - I_1(2\mu r)] + r \right) \right\}$$

which reduces to

$$1/c = \frac{1}{r\mu} [I_1(2\mu r) + L_1(2\mu r)]$$

The functions $I_1(2\mu r)$ and $L_1(2\mu r)$ may be evaluated by programs written for the MONROE 1655 programable desk calculator.

For the capsules in question the results were

$$r = 0.269 \text{ in.}$$

$$\mu = 2.9355 \text{ cm}^2/\text{g}.$$

$$L_1 = 0.6238$$

$$I_1 = 1.0628$$

$$1/c = 0.5559$$

A-1

ANC-A-27

FIGURE A 1. Sectional view of the sample.

FIGURE A 2. An element of area
shown in A 1.