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DOE RESEARCH AND  
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MASTER

# THE NONDESTRUCTIVE ASSAY OF UO<sub>2</sub>-ThO<sub>2</sub> FUEL PELLETS USING THE DELAYED NEUTRON PELLET ASSAY GAGE

(LWBR Development Program)

CP JUNE 1979

CONTRACT EY-76-C-11-0014

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(LWBR Development Program)

C. J. Emert, S. Milani, and W. J. Beggs

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## FOREWORD

The Shippingport Atomic Power Station located in Shippingport, Pennsylvania was the first large-scale, central-station nuclear power plant in the United States and the first plant of such size in the world operated solely to produce electric power. This program was started in 1953 to confirm the practical application of nuclear power for large-scale electric power generation. It has provided much of the technology being used for design and operation of the commercial, central-station nuclear power plants now in use.

Subsequent to development and successful operation of the Pressurized Water Reactor in the DOE-owned reactor plant at the Shippingport Atomic Power Station, the Atomic Energy Commission in 1965 undertook a research and development program to design and build a Light Water Breeder Reactor core for operation in the Shippingport Station.

The objective of the Light Water Breeder Reactor (LWBR) program has been to develop a technology that would significantly improve the utilization of the nation's nuclear fuel resources employing the well-established water reactor technology. To achieve this objective, work has been directed toward analysis, design, component tests, and fabrication of a water-cooled, thorium oxide fuel cycle breeder reactor for installation and operation at the Shippingport Station. The LWBR core started operation in the Shippingport Station in the Fall of 1977 and is expected to be operated for about 3 to 4 years. At the end of this period, the core will be removed and the spent fuel shipped to the Naval Reactors Expended Core Facility for a detailed examination to verify core performance including an evaluation of breeding characteristics.

In 1976, with fabrication of the Shippingport LWBR core nearing completion, the Energy Research and Development Administration established the Advanced Water Breeder Applications (AWBA) program to develop and disseminate technical information which would assist U. S. industry in evaluating the LWBR concept for commercial-scale applications. The program will explore some of the problems that would be faced by industry in adapting technology confirmed in the LWBR program. Information to be developed includes concepts for commercial-scale prebreeder cores which would produce uranium-233 for light water breeder cores while producing electric power, improvements for breeder cores based on the technology developed to fabricate and operate the Shippingport LWBR core, and other information and technology to aid in evaluating commercial-scale application of the LWBR concept.

## FOREWORD (Cont)

All three development programs (Pressurized Water Reactor, Light Water Breeder Reactor, and Advanced Water Breeder Applications) have been administered by the Division of Naval Reactors with the goal of developing practical improvements in the utilization of nuclear fuel resources for generation of electrical energy using water-cooled nuclear reactors.

Technical information developed under the Shippingport, LWBR, and AWBA programs has been and will continue to be published in technical memoranda, one of which is this present report.

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This report describes the use of a delayed neutron pellet assay gage to determine nondestructively the fissile content of fuel pellets during the manufacture of the Light Water Breeder Reactor (LWBR) core. The gage characteristics are described including the nature of the calibration curves and the gage sensitivities to pellet parameters. Statistical methods are derived for analyzing the data to obtain the mean weight percent of total uranium in each blend of fuel material as well as the loading precision of each fuel rod. The fissile loading of each fuel rod was determined to better than 0.25% at the  $2\sigma$  level, and the fissile content of eight fuel compositions in the LWBR core was obtained to better than 0.1%. Use of this gage and the data analysis methods described in this report reduced the need for destructive chemical analysis of fuel pellets by a factor of two.

THE NONDESTRUCTIVE ASSAY OF  $\text{UO}_2\text{-ThO}_2$  FUEL PELLETS  
USING THE DELAYED NEUTRON PELLET ASSAY GAGE  
(LWBR Development Program)

C. J. Emert, S. Milani, and W. J. Beggs

I. INTRODUCTION

The Light Water Breeder Reactor (LWBR) has a seed-blanket core configuration using  $^{233}\text{U}$  as the fissile fuel and thorium as the fertile fuel. The core consists of an inner region containing 12 movable seed assemblies, each surrounded by a blanket assembly, and an outer region containing 15 reflector modules. Contained in the core are three different diameter  $^{233}\text{UO}_2\text{-ThO}_2$  binary fuel pellets and four different diameter  $\text{ThO}_2$  reflector fuel pellets. The binary pellets were manufactured in a total of eight fuel types with six significantly different weight percents of total uranium. Tables 1A and 1B present the physical attributes and isotopic compositions of these pellets. For binary pellet fabrication, a specified amount of uranium oxide was mixed with thorium oxide powder. The combined mixture was called a blend. There were approximately 900 blends produced during binary fuel pellet manufacture.

To evaluate the ability of the LWBR core to meet its design objective of breeding, it is necessary to determine accurately both the beginning-of-life and end-of-life inventory of fissile material in the fuel. The delayed neutron pellet assay gage was developed to provide an instrument of high precision which could

TABLE 1A. LWBR  $^{233}\text{UO}_2\text{-ThO}_2$  PELLET PHYSICAL ATTRIBUTES\*

<u>Pellet Description</u>	<u>Weight (gm)</u>	<u>Length (cm)</u>	<u>Diameter (cm)</u>	<u>Density (gm/cm<sup>3</sup>)</u>	<u>Nominal **w/o <math>\text{TU}</math></u>
Low zone seed	3.502	1.1285	0.6401	9.804	4.408
High zone seed	4.864	1.5618	0.6401	9.795	5.285
Low standard blanket	17.161	1.3480	1.2967	9.872	1.234
Medium standard blanket	28.147	2.2047	1.2967	9.807	1.695
High standard blanket	25.482	1.9949	1.2967	9.827	2.038
Low power flattening blanket	23.883	2.2088	1.1925	9.817	1.681
Medium power flattening blanket	21.558	1.9959	1.1925	9.820	2.044
High power flattening blanket	19.191	1.7810	1.1928	9.812	2.791

\*Averages of delayed neutron gage sample pellets from blends used in the LWBR core.

\*\*Percent of pellet weight contributed by  $\text{TU}$  (total uranium).

TABLE 1B. NOMINAL ISOTOPIC COMPOSITION\*

<u>Pellet Description</u>	<u><math>^{233}\text{U}</math></u>	<u><math>^{234}\text{U}</math></u>	<u><math>^{235}\text{U}</math></u>	<u><math>^{236}\text{U}</math></u>	<u><math>^{238}\text{U}</math></u>
Low zone seed	98.299	1.294	0.083	0.019	0.305
High zone seed	98.367	1.275	0.068	0.014	0.275
Low standard blanket	98.303	1.329	0.088	0.024	0.254
Medium standard blanket	98.319	1.306	0.081	0.020	0.273
High standard blanket	98.254	1.354	0.107	0.031	0.254
Low power flattening blanket	98.310	1.319	0.086	0.022	0.263
Medium power flattening blanket	98.210	1.372	0.119	0.035	0.265
High power flattening blanket	98.040	1.243	0.097	0.026	0.593

\*Percent of uranium by weight. Data are averages of sample pellets which were analyzed by mass spectroscopy and represent blends used in the LWBR core.

determine nondestructively the fuel content and macroscopic homogeneity of fuel pellets manufactured for the LWBR core. The use of this gage during manufacturing operations resulted in a reduction by a factor of two in the amount of chemical assay that was required, and its use improved the precision of the fissile fuel analysis.

There were two main requirements that the delayed neutron pellet assay gage had to satisfy. First, the gage together with the concurrent chemical analysis had to provide the fissile loading of each fuel rod with a precision of 0.25% at the  $2\sigma$  level. This fuel rod loading precision was chosen to support the 0.1% precision at the  $2\sigma$  level needed in each of the eight fuel zones (Tables 1A and 1B) to evaluate the core against its breeding objective. The fuel rod loading precision requirement was met by assaying one pellet at a time with a random sample of 20 pellets per blend in a prescribed sequence with the delayed neutron pellet assay gage and 5 of these 20 pellets by chemistry. The 20 pellets represented the 1,000 to 5,000 pellets from each blend which were used in the core. The prescribed sequence for gage assay included repetitions of the 20 pellets and other pellets used to calibrate and standardize the gage operation. As a corollary to the precision requirement, the gage assay yielded an estimate of the pellet-to-pellet variability of the uranium content.

The second main requirement which the gage had to satisfy was to support the schedule of pellet manufacturing and core assembly. The assay of a blend by the gage and by chemistry had to be complete before fuel rods containing that blend could be used to construct a seed or blanket assembly. This requirement was met by the gage without compromising the precision requirement by assaying about 1-1/2 blends per eight-hour shift.

In attaining the required precision, it was desired to minimize the amount of chemical analysis performed because of the significant cost and time required for chemical analysis. The gage with 20 samples per blend had better precision than chemistry with 5 samples per blend. Without the gage, the chemistry effort would have at least doubled to obtain the same precision as the combination of gage and chemistry.

Delayed neutron assay of samples containing fissile material has been successfully used in the past for a variety of applications (References 1 and 2). The basic technique uses a source of neutrons to induce fissions in a sample that

contains fissile fuel. Delayed neutrons that result from fission product decay are subsequently emitted from the sample. The number of delayed neutrons emitted depends upon the number of fissions that occurred and hence upon the amount of fissile fuel in the sample. Early experiments (Reference 3) were conducted to test the feasibility of constructing a high precision fuel pellet assay device. The outcome of these experiments identified possible problem areas. One of the problems was the inefficiency of manual gage operation, and this was resolved through automation. Another problem, corrosion of the main structural pipe in the gage, was solved by use of sufficiently heavy stock. High background counts, caused by the neutron sources used as part of the gage, were reduced by distance and shield material. The design of the delayed neutron pellet assay gage successfully solved these problems, as discussed in subsequent sections. In addition, equations for the statistical analysis of the gage error and the combination of gage and chemistry results are presented.

Prior to routine operation of the gage, it was necessary to obtain a calibration curve of counts per gram of pellet versus the weight percent of uranium in the pellet for each of the three diameters of fuel pellets. This was done by determining the response of the gage for 30 randomly selected pellets from one "calibration blend" for each fuel type and then performing chemical analysis on 20 of these pellets. The remaining 10 pellets were called "retainer samples". The gage response was sensitive to variations in diameter, length, and density of the pellets, and corrections were made using measured sensitivities. The corrected data were least squares fitted to a quadratic function to obtain the calibration curve and its associated error.

For subsequent blends 20 pellets per blend were measured in the gage, together with retainer samples from the calibration blend, to obtain a gage value of the blend uranium weight percent and the pellet-to-pellet variability in the weight percent. Five of these 20 pellets were subsequently chemically analyzed. The gage and chemistry results were combined to obtain a maximum likelihood estimate of the blend mean weight percent of total uranium. This value plus the weight of the pellets from that blend which were loaded into a fuel rod were used to obtain the contribution of that blend to the fuel rod loading. For each of the fuel rods contained in the LWBR core, the rod loading precision was calculated to be 0.25% or better at the 95% confidence level.

Section II provides a brief description of the gage, and Section III describes the operation and data reduction methods. Results of the gage sensitivity studies are presented in Section IV. Section V describes the methods used to obtain the calibration curves and a statistical derivation of the calibration error. Section VI presents the method used to obtain the gage value of the blend weight percent and the statistical methods derived to obtain its error. In Section VII, the method used to combine the gage and chemistry values and the derivation of the maximum likelihood estimator of the blend mean are presented. Also the method of obtaining the error on the mean is derived. Conclusions are presented in Section VIII.

## II. DESCRIPTION OF THE GAGE

The essential features of the delayed neutron pellet assay gage are shown in Figure 1. They consisted of a 19,000 liter shield tank, two large neutron sources and six  $^3\text{He}$  neutron detectors located in the tank, a glove box for handling and encapsulating the fuel pellets, and a means of sample transport. The shield tank, which was outdoors and filled with water, was thermally insulated and electrically heated to maintain a controlled, uniform temperature. An aluminum pipe, 15.2 cm in diameter, extended the length of the tank and penetrated the building wall. Within this pipe were water extended polyester cylinders into which two 5 mg  $^{252}\text{Cf}$  spontaneous fission neutron sources and six  $^3\text{He}$  detectors were inserted. The neutron sources were located near the middle of the tank, and the detectors were located near one end of the tank. The  $^3\text{He}$  detectors were chosen because of their high thermal neutron sensitivity.

The encapsulated sample was transported on an 8.5 meter cog timing belt driven by a stepping motor. The sample was transported from a glove box located inside of the building to a position near the detectors to obtain a background count. At the completion of the background count, the sample was transported close to the sources for irradiation and then subsequently back to the count position. After the completion of the delayed neutron count, the sample was returned to the glove box. The sample was transported at speeds of 60 cm per second inside a 4.4 cm x 10.2 cm O.D. rectangular aluminum tube within the 15.2 cm diameter pipe. The sample remained in the irradiation position and in the count position the same length of time. Although the count and irradiation times were initially 100 seconds, they were reduced later to 50 seconds for most of the fuel pellet assays.

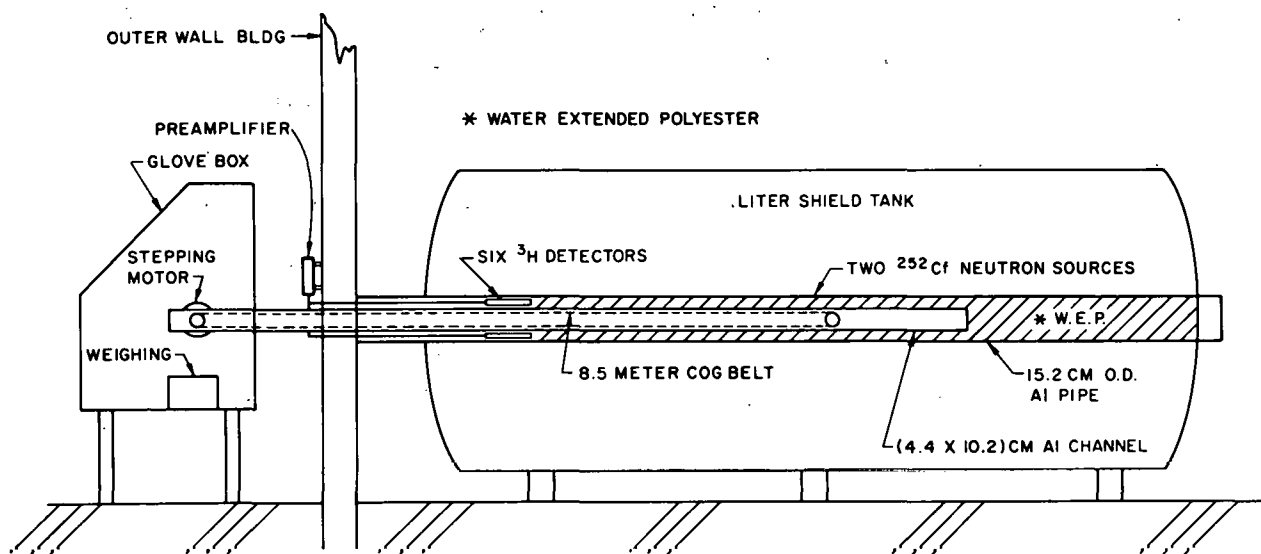


Figure 1. LWBR Pellet Assay Gage

Several problems were overcome during the development of the gage. The central aluminum pipe which extends through the shield tank is an anodized pipe about 6 meters long with 0.64 cm wall thickness. This relatively heavy stock was used to overcome corrosion of the pipe. Similarly, rubber O-rings were used to complete the seal where the central aluminum pipe passes through the iron ends of the shield tank. This avoided corrosion due to the dissimilar metals.

High detector count rates due to source neutrons diffusing through the shield tank water were avoided by keeping the sources and detectors about 2-1/2 meters apart. A 76 cm long lucite bar was made to follow behind the sample holder and was always between the sources and detectors. The bar scattered source neutrons streaming down the open channel within which the sample was transported.

Reference 3 contains a detailed description of the construction of the gage and of the electronics. Two modifications were incorporated into the gage design after Reference 3 was published. The first modification was to incorporate an automatic sample changer into the gage operation. The sample changer duplicated the manner in which a blend of pellets was manually processed through the gage. Each blend contained 20 pellets to be assayed by the gage. The automatic sample changer could accommodate 20 pellets from each of three blends in a single loading. In addition to the blend samples, the changer could also accommodate three retainer samples from the calibration experiment and a high count rate standard.

Retainer samples were used for normalization purposes, while the high count rate standard was used to correct the data for drifts in the electronics. High count rates provided good statistical precision in a short time. A regular blanket-sized  $^{235}\text{UO}_2\text{-ThO}_2$  pellet containing 1.5 grams of  $^{235}\text{U}$  was used as the high count rate standard.

To ensure that the changer had operated properly, several counters were used to monitor different aspects of the changer operation. One counter advanced each time the changer would step from one pellet position to the next position. This counter had a provision to preset the number of steps that would be required for the entire assay. When the number of steps advanced reached the preset value, the counter would terminate the operation of the gage. A second counter was used to indicate the number of blends that were assayed, i.e., one, two, or three. This counter was useful in determining the status of the changer operation.

The second modification was to incorporate a minor change into the output data format of a teletype scanner. The function of this unit was to accept the delayed neutron count at the end of a preset count time and to transmit this information to a teletype terminal where it was printed and also punched onto paper tape. To preclude the operator from entering arbitrary count data onto the teletype, the output of each line of data from the scanner was concluded with a coded character which could not be entered from the teletype keyboard. This character was subsequently acknowledged by the analytical code that interpreted the count data from the gage. If the coded character was missing, an appropriate message was printed to alert the data reduction personnel of a possible error.

### III. GAGE OPERATION

#### A. Summary

A summary of the typical sequence of events governing the operation of the gage is as follows. After the weight, length, and diameter of the pellets from the blend to be assayed were determined, they were placed into cylindrical Zircaloy capsules and then loaded into the automatic sample changer. The sample holder attached to the timing belt was checked to make sure that the starting position was correct, the counting channels were checked for proper operation, the sample changer was set for the correct number of operations to be performed based upon the number of samples to be assayed, and all counters and stepping relays were reset to their initial conditions. Depressing the "start" switch



loaded the first sample into the sample holder attached to the timing belt and started the automatic sequencing of the gage. For each sample, a background and delayed neutron count were obtained. All of the counts were accumulated for the same period of time, i.e., 50 seconds. After the completion of the delayed neutron count, the sample was returned to the glove box and the counting cycle was repeated for the next sample. At the completion of the selected number of pellet operations, the changer automatically stopped the assay. The samples were then removed from the automatic changer and returned to their containers. This summarizes a typical operating sequence for the gage. Each of the steps outlined above will now be considered in greater detail.

#### B. Initial Data

Each pellet was inspected for proper identification symbols pressed into the top and bottom of the pellet during manufacture, the pellet length and diameter were measured, and it was weighed. The dimensions were recorded to the nearest 0.00025 cm, and the weights were recorded to the nearest 0.1 milligram.

The identification of the pellets to be assayed, the capsules into which these pellets were loaded, and the position of these capsules in the automatic sample changer were recorded. The identities of special retainer samples that were used to normalize the data to the calibration experiment were also recorded along with the identity of a high count rate standard. The proper position of the sample holder attached to the timing belt was determined by the use of a gage block.

#### C. Operational Checks

A daily statistical check was performed for each of two counting channels. Each channel totaled the counts from three of the  $^3\text{He}$  detectors. The check consisted of counting the background 10 times on each channel with a pellet of the type to be assayed located in the background count position. The average background count and its standard deviation were calculated for each channel. The observed standard deviation was required to be within the range  $0.302 \sigma \leq s \leq 1.644 \sigma$ , where  $\sigma$  was taken to be the square root of the average background count. These unbiased s-chart control limits were obtained from Reference 4. If this check was unsatisfactory, which might for example indicate excessive noise or the missing of counts, then it was repeated two additional times. If the latter two checks were satisfactory, indicating that the previous result was a chance

fluctuation, then operations could proceed. If either one of the latter two checks was not satisfactory, then operations were suspended pending resolution of the problem.

Checks were also made on the background counts obtained during the assay of each blend. Limits were established which required that the background count be within  $\pm 4.433 \sigma$ . The factor 4.433 is the 95%/99% tolerance value. The 20 pellets from the blend were reassayed if more than two background counts exceeded these limits.

The 20 pellets per blend that were assayed by the gage were counted three times each. Three counts were the minimum number of counts which could be used to determine statistically if an outlier existed in any of the pellet count data. The method used to detect and replace outliers in the data is outlined in the Appendix. (An outlier is defined as an observation far removed from the main body of the data.)

#### D. Counting Method

The assay gage samples were counted in the following manner. The first sample changer position contained a high count rate standard. The assay of this pellet, designated STDA, produced a large delayed neutron count, i.e., 500,000 counts or more. STDA was counted before and after each block of 10 pellets from the blend being assayed. The purpose of counting this pellet was to provide a way to correct the count data for any possible drift that might occur during the counting of a single blend as a result of room temperature changes, changes in the electric line voltage, etc. The next three sample changer locations were used for retainer pellets. The retainer samples were fuel pellets from the calibration blend.

The retainer pellets were recounted after each block of 10 pellets from the blend being assayed. The retainer pellets provided the means by which the data from the blend being assayed could be normalized to the calibration experiment. The next 20 locations were reserved for pellets from the first blend to be assayed. The changer could accommodate 20 pellets from each of three blends.

Depressing the start switch initiated the sample analysis by loading STDA into the sample holder attached to the timing belt. The pellet was then transported to the background count position. At the completion of the background

counting interval (50 seconds), STDA was then moved to the irradiate position near the  $^{252}\text{Cf}$  sources. At the completion of the 50 second irradiation time, a count was printed for each channel. This irradiate count was not used directly in the analysis of the data; however, it was of interest because it did not vary greatly from pellet to pellet, and it was useful in diagnosing problems in the gage data. At the completion of the irradiation time, STDA was returned to the counting position and the induced delayed neutron count was obtained. It required four seconds to travel from the irradiate position to the counting position. At the completion of the delayed neutron count, STDA was returned to the glove box position where it was ejected from the sample holder and loaded back into the automatic sample changer. The sample changer then advanced to the first retainer pellet and the same assay sequence was repeated. The next two retainer samples and the first 10 pellets of the blend were then assayed. After the tenth blend pellet, STDA and the three retainer pellets were counted again. Following the counting of the retainer pellets, samples 11 through 20 of the blend were assayed. At the completion of pellet 20, STDA and the three retainer pellets were counted for the third time. The entire assay sequence, STDA, R1, R2, R3, Pellets 1 through 10, STDA, R1, R2, R3, Pellets 11 through 20, STDA, R1, R2, R3, was repeated three times for each blend of pellets to be assayed. At the completion of the first blend of 20 pellets, and if the assay were to continue, the sample changer advanced to the second blend of 20 samples and proceeded with the same order of pellet counting as for the first blend of samples. At the completion of the desired number of assays, the changer automatically stopped the gage operation.

The assay gage operator, prior to unloading the samples from the sample changer, would check to see that the gage had operated properly. Items checked included seeing that the changer had indexed the correct number of steps, that the changer had stopped at the correct position, and that the counter indicated the correct number of unknown blends of pellets that were to be assayed. The teletype output was checked for abnormal data and the position of the sample holder was again checked with the gage block to see that it had not changed. If the above items were satisfactory, then the samples that were assayed were unloaded from the sample changer, removed from their capsules and placed back into their

storage container. The identity of each pellet was retained throughout the handling operations. Successful completion of the above checks was indicated on the sample changer loading form.

#### E. Performance Checks

Three checks were used to assure that the gage was performing properly. The first check required that the average relative bias between the gage and the destructive chemical analysis for 25 pellets be less than 0.15%. The 25 pellets were the first five pellets of each blend for the first five blends following the calibration blend. The second check required that the difference between the gage and chemistry estimates of the weight percent of total uranium for a given blend be no greater than a specified limit. This limit was somewhat different for each blend that was analyzed because it took into account the experimental uncertainty in both the chemistry data and in the assay gage data. The third check required that if the sign of the difference between the gage and chemistry results were the same for nine blends in a row, then an average bias correction would be applied to the gage data based upon the average bias of these nine blends. The basis for these checks is developed in Section VII.

#### F. Operating History

Over 19,000 pellets were processed through the gage during the period of 2.7 years that the gage was used for production pellet assay. Prior to the start of production assay, preliminary measurements were conducted for a period of approximately one year. The normalization of the count rate data using the retainer samples takes into account the effect of decay of the  $^{252}\text{Cf}$  source strength. The neutron sources were replaced midway through the period of usage.

Throughout the entire period of use, the electronics performed well. The sample transport mechanism, consisting of the stepping motor, indexer, and cog timing belt, performed flawlessly throughout the entire assay period. There was a minimal amount of system downtime, the longest period being a week required for the installation of the automatic sample changer.

### IV. GAGE SENSITIVITIES TO PHYSICAL PARAMETERS

The sensitivities of the gage response, in terms of counts per gram of pellet, to variations in gage parameters and pellet attributes were measured. The use of retainer pellets to normalize the results to the calibration blend eliminated any

bias due to gage parameter variations, so that the gage parameter sensitivities were not directly used in the calculations. The gage parameter sensitivities were used to judge their own importance and thereby influenced the design of the gage. The pellet attribute sensitivities, on the other hand, were used directly in adjustments of the data. The results of the sensitivity studies are given in the following sections.

#### A. Effect of Variations in Pellet Positioning

The effects of variations in pellet positioning at the detector and source irradiation locations were measured. Figures 2 and 3 show the change in response when the pellet locations were varied by 5 or 10 steps at a time in the vicinity of the source and detector locations, respectively. The arrows in Figures 2 and 3 show the positions where the pellet was placed for irradiation and for counting. Each step represents a 0.0805 cm displacement of the pellet. The sensitivity to positioning at the source location was 0.011% change in sample counts per step or 0.138% change in sample counts per cm displacement of the pellet. At the detector location, the sensitivity was 0.095% per step or 1.181% per cm of displacement.

#### B. Temperature Effects

##### 1. Room Temperature Changes

The change in response of the gage with room temperature was measured as the room temperature increased from 21.7°C to 26.1°C. A proportional temperature controller was used to maintain a stable discriminator and preamplifier ambient temperature during these tests. A linear least squares fit to these points gives a sensitivity of +0.103% change in response per degree Celsius. A second set of data was obtained on a different day covering the interval 23.9°C to 27.5°C. The sensitivity determined from these data was +0.095% change in response per degree Celsius.

##### 2. Instrument Temperature Sensitivity

a. The preamplifier temperature sensitivity was observed to be +0.047% per degree Celsius in the range of 21.2°C to 25.0°C. This effect was measured by heating the preamp electrically with the preamp temperature controller disabled, while maintaining the remainder of the system at constant temperature.

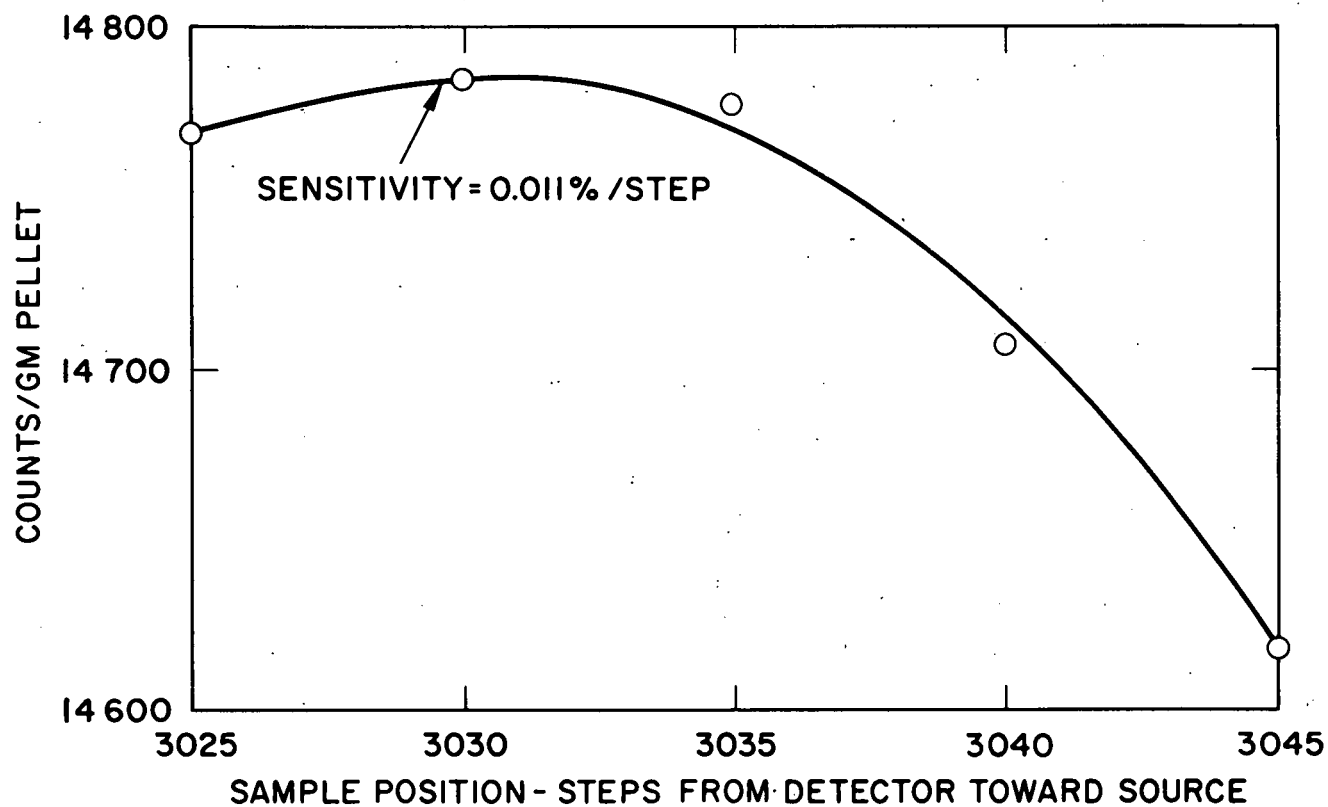


Figure 2. Position Sensitivity at Source Location

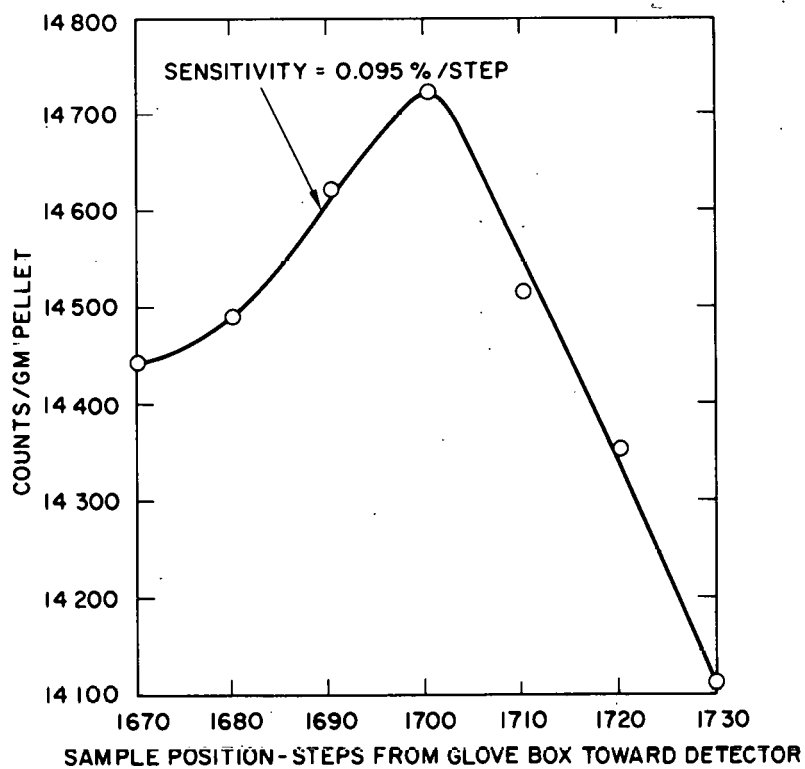


Figure 3. Position Sensitivity at Detector Location

b. The discriminator temperature sensitivity was determined to be 0.155% per degree Celsius in the range of 20.6°C to 25.4°C. This sensitivity was measured with the proportional temperature controller on the discriminator disabled.

### 3. Tank Temperature Effects

The temperature of the water in the shield tank was observed to affect the results. This sensitivity was measured by repeat measurements of retainer pellets over a 5.6°C range of water temperatures. The results yielded a sensitivity of 0.14% per degree Celsius. To minimize this effect, heating elements and provision for water circulation were added to the tank so that temperature fluctuations could be controlled to within a few degrees.

### C. Effect of Variability in Pellet Dimensions and Density

The sensitivities to pellet length, diameter, and density were measured. Sensitivities measured using  $^{235}\text{U}$  pellets were compared in a few cases with those using  $^{233}\text{U}$  pellets. These measurements confirmed the theory that if adjustments were made to take into account the relative neutron absorption in the pellets, the sensitivity measured using  $^{235}\text{U}$  data could be used for  $^{233}\text{U}$  pellets. The following types of fuel pellet materials were used to evaluate the effects of pellet dimensions, density, and isotope on the gage response.

<u>Composition</u>	<u>Diameter Range (cm)</u>	<u>Length Range (cm)</u>	<u>Theoretical Density (%)</u>
1.99 w/o $^{\text{T}}\text{U}$ * in $\text{UO}_2\text{-ThO}_2$ (Uranium enriched to 93.14% $^{235}\text{U}$ )	0.640-1.339	1.118-2.540	94.00
1.99 w/o $^{\text{T}}\text{U}$ in $\text{UO}_2\text{-ThO}_2$ (Uranium enriched to 93.14% $^{235}\text{U}$ )	0.640-1.339	1.118-2.540	97.80
4.39 w/o $^{\text{T}}\text{U}$ in $\text{UO}_2\text{-ThO}_2$ (Uranium enriched to 97.96% $^{233}\text{U}$ , 0.216% $^{235}\text{U}$ )	0.564-0.640	1.130	97.01
5.52 w/o $^{\text{T}}\text{U}$ in $\text{UO}_2\text{-ThO}_2$ (Uranium enriched to 96.51% $^{233}\text{U}$ , 0.629% $^{235}\text{U}$ )	0.637	0.889-1.854	95.63

\*Throughout this report,  $^{\text{T}}\text{U}$  is the symbol used for total uranium.

### 1. Diameter Sensitivity

Figure 4 summarizes the sensitivity of the gage to pellet diameter as a function of pellet length for diameter sizes covering the diameter range from seed to standard blanket size pellets. These curves were obtained using the 1.99 w/o  $^{235}\text{U}$ , low density pellets (94 percent of theoretical density) which were precision ground to specified lengths and diameters. Prior to precision grinding, these pellets were selected from a group of 50 blanket pellets which had been previously examined with the pellet assay gage. The gage measurements showed that this group of pellets was very homogeneous, having a standard deviation of approximately 0.08% in counts per gram after taking into account length, diameter, and density effects but including the gage error. Using an estimate of the gage error of 0.05% based on other data implies a pellet-to-pellet variability of approximately 0.06%. Since the variations observed in the diameter sensitivity measurements were large compared with the pellet-to-pellet variability, the results accurately reflect the effects of changes in the diameter. The diameter sensitivity was negative, i.e., the larger the diameter the smaller the counts per gram of pellet.

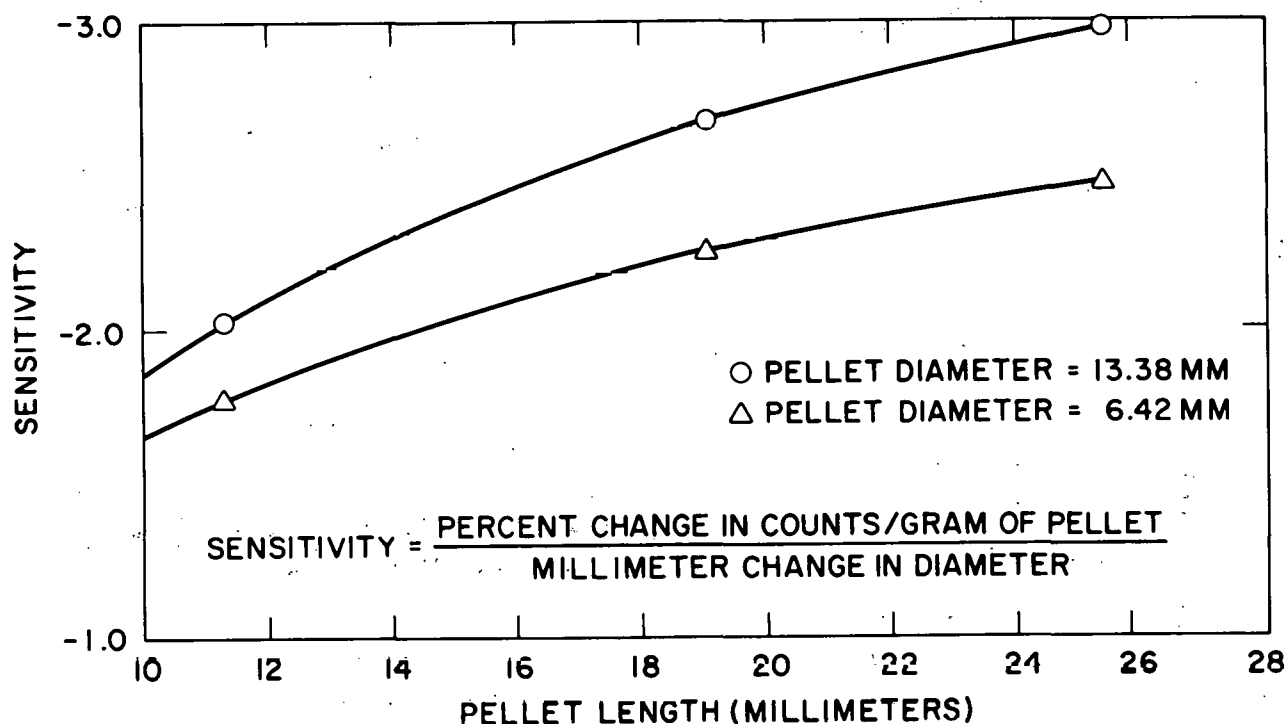


Figure 4. Diameter Sensitivity versus Length



## 2. Length Sensitivity

Figure 5 shows the pellet length sensitivity of the gage as a function of pellet length. Results showing the effect of diameter on length sensitivity are also included. These measurements were obtained from precision ground pellets taken from the same group of 50 1.99 w/o  $TU$  pellets described in the previous section. The sign of these sensitivities is also negative.

## 3. Density Sensitivity

Figures 6 and 7 show the effect of length and diameter on the density sensitivity which is defined as the percent change in counts/gram of pellet per percent change in pellet density. The sign of the density sensitivity is also

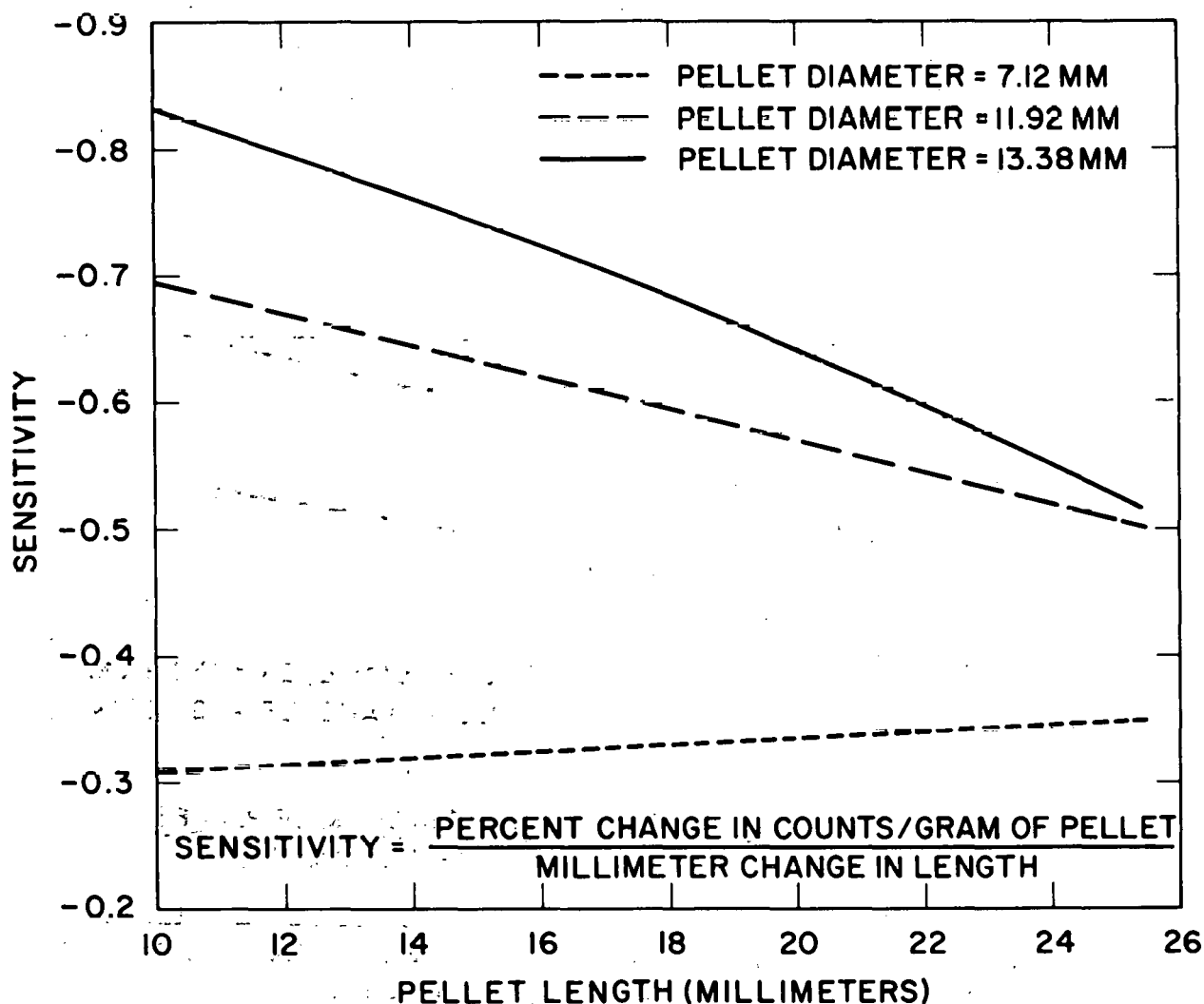


Figure 5. Length Sensitivity versus Length

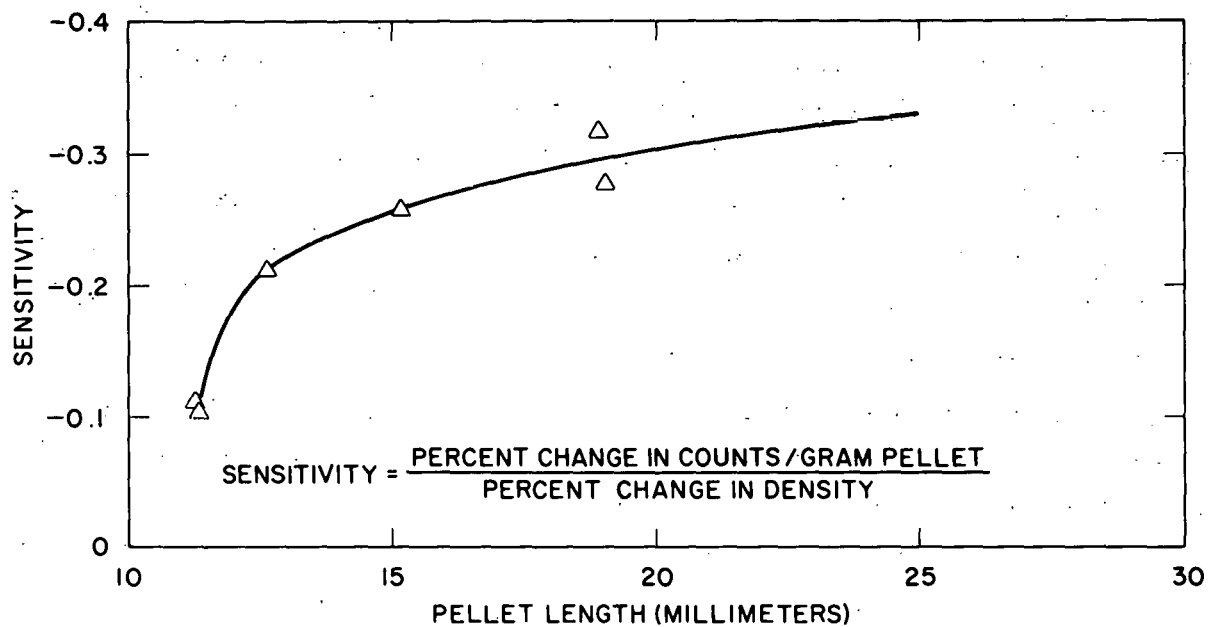


Figure 6. Density Sensitivity versus Pellet Length

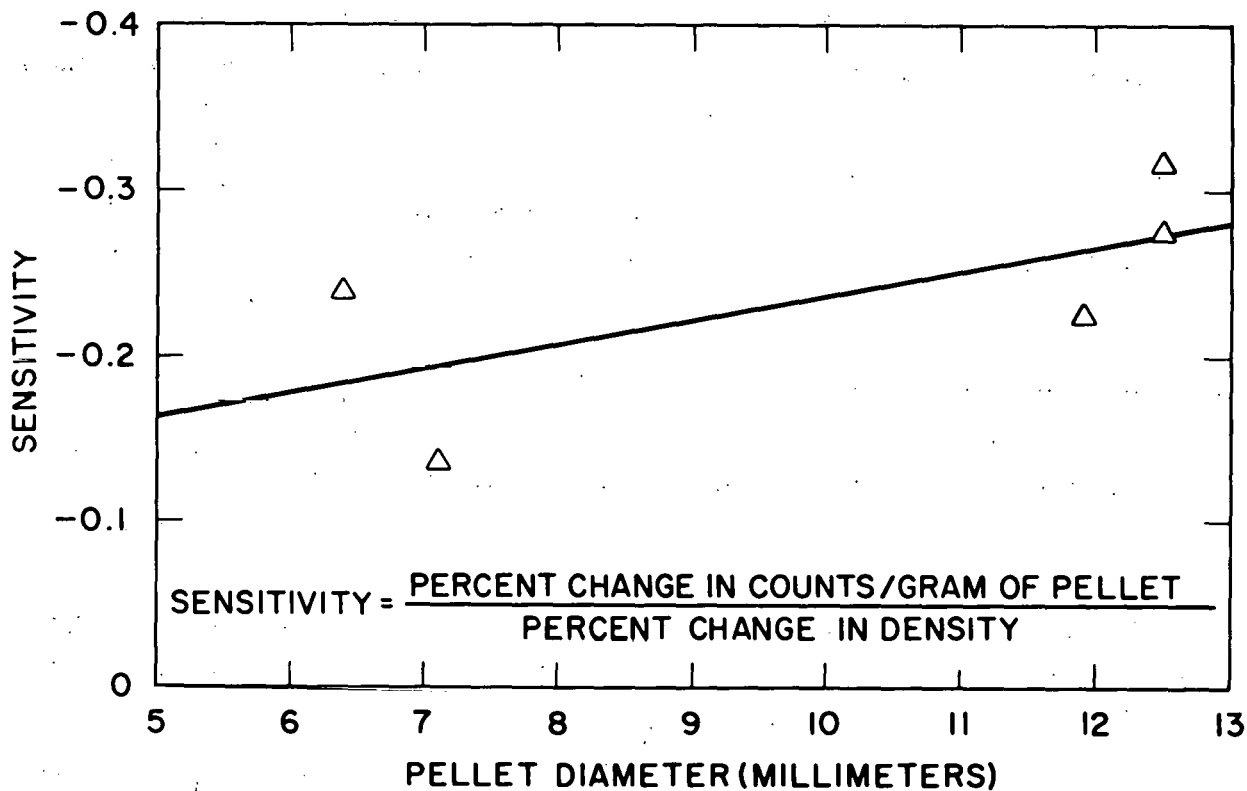


Figure 7. Density Sensitivity versus Pellet Diameter

negative. These data were obtained using precision ground pellets from the same group of 50 low density pellets plus additional 1.99 w/o  $^{235}\text{U}$  precision ground pellets made from the same powder lot as the low density pellets but which were sintered to 97.8% instead of 94% of theoretical density. The theoretical density for these pellets is 10.023 grams per cubic centimeter. Pellet gage measurements of other high density pellets from the same lot used to make the high density precision ground pellets gave a standard deviation of approximately 0.04%, indicating good homogeneity of pellets from this lot.

The data show greater scatter than was observed in previous sensitivity measurements. This may be due to the higher error associated with the smaller differences in response observed between pairs of samples. The results indicate that the density sensitivity increased with the increasing pellet diameter and pellet length although the scatter in the data obscures some of the effects.

#### 4. Chip Sensitivity

Chip sensitivity measurements were performed using the low density standard blanket size pellets of 1.99 w/o  $^{235}\text{U}$ . For a chip representing a 1.12% change in the pellet volume, the change in the counts per gram of pellet increased by 0.21%. Similarly, a chip corresponding to 5.2% of the pellet volume increased the counts per gram of pellet by 1.52%. The average effect is  $(0.24\% \Delta y/y / \% \Delta v/v)$  where  $y$  is the counts per gram and  $v$  is the pellet volume. These data were not used directly in calculations; they were only for information. To use these results with other pellet types, the data must be corrected for relative weight percent, absorption cross section, and for the slope of the calibration curve.

#### 5. Relative $^{233}\text{U}$ and $^{235}\text{U}$ Sensitivity Response

Most of the sensitivities were measured with  $^{235}\text{U}$  pellets, since  $^{233}\text{U}$  pellets were not available. When  $^{233}\text{U}$  seed pellets became available, the diameter and length sensitivities were measured and compared with the values obtained from the 1.99 w/o  $^{235}\text{U}$  pellets. The data showed that the sensitivities for the  $^{233}\text{U}$  pellets differed from the  $^{235}\text{U}$  data. The difference for the same diameter pellet is believed to be due primarily to the difference in the macroscopic absorption cross sections of the two types of pellets.

The diameter sensitivity of seed size binary preproduction  $^{233}\text{U}$  pellets of 4.39 w/o  $^{235}\text{U}$  was obtained by using normal diameter pellets and 10 similar pellets

which were ground undersize. This resulted in a diameter sensitivity of  $-(3.2 \pm 0.6)\%/mm$  at an average diameter of 0.6 cm. For the low density  $^{235}U$  pellets having 1.99 w/o  $^{235}U$ , a comparable sensitivity is  $-(1.7 \pm 0.3)\%/mm$ . Here the ratio of the sensitivities ( $1.9 \pm 0.5$ ) is approximately equal to the ratio of the macroscopic absorption cross sections of the fissile materials (2.0).

The counts per gram as a function of length for  $^{233}U$  pellets having 5.52 w/o  $^{233}U$  and a 0.637 cm diameter were measured. The length sensitivity for 1.27 cm long pellets was observed to be  $-(0.39 \pm 0.08)\%/mm$ . This is to be compared to  $-(0.25 \pm 0.04)\%/mm$  for the  $^{235}U$  pellets. The ratio of sensitivities ( $1.6 \pm 0.4$ ) is within two standard deviations of the ratio of the macroscopic absorption cross sections (2.4).

For pellets of a given diameter, sensitivities measured with  $^{235}U$  pellets were applied to the  $^{233}U$  pellets by first correcting for the difference in the macroscopic cross sections.

#### 6. Chamfer Sensitivity

The sensitivity of the seed pellet response to a change in 45-degree pellet chamfer was measured to be 1.1% and 0.6% change in the counts per gram of pellet per mm change in pellet chamfer for the low zone and the high zone seed pellets, respectively. These sensitivities were determined from measurements of the counts per gram of pellet both before and after chamfers were applied to the same set of pellets.

### V. CALIBRATION CURVE

#### A. Determination of the Calibration Curves

A separate calibration curve was calculated for each of the eight types of pellet, or pellet compositions, given in Tables 1A and 1B. Each calibration curve consisted of a weighted least-squares quadratic fit of the counts per gram of pellet to the pellet weight percent of total uranium. To construct a typical calibration curve for any one composition, data for calibration blends from three compositions were included in order to cover a range of weight percent of total uranium. The two compositions in the calibration curve, other than the one for which the curve was being generated, served to determine the slope of the fitted curve near the composition of interest (calibrating composition). Twenty pellets per composition for each of the three compositions were used and each pellet was counted five times.

The data for the two compositions in the calibration curve other than the calibrating composition were adjusted for any differences in length, diameter, and density to those of the calibrating composition. The length adjustment was made, for example, because the pellets of different compositions had different pellet lengths and the gage was sensitive to changes in the pellet length. The diameter adjustment was small because only pellet types with the same nominal diameter were used in a given calibration curve.

Typical calibration curves for the seed and standard blanket pellets are shown in Figures 8 and 9. For the calibration curves of the low zone compositions for each nominal diameter of pellet, thorium pellets were assayed to provide the third composition, i.e., zero weight percent of total uranium. Each point on the figures represents a cluster of 20 pellets for non-zero w/o  $^{235}\text{U}$  or a cluster of 10 or fewer pellets of thorium. During the initial manufacturing operations, pellets of all compositions were not available. Therefore, pellets produced during preproduction manufacturing operations were used in the calibration curves.

A typical gage calibration experiment consisted of assaying each of 30 pellets from a single blend five times. Twenty of the 30 pellets assayed by the gage were also analyzed by chemistry. Of the 10 remaining pellets, typically three were selected to be used as retainer samples. These retainer samples were counted every time a blend of pellets of unknown weight percent uranium, but whose composition was nominally the same as the pellets of the calibrating composition, was assayed by the gage. The ratio of the counts produced by the retainer samples at the time a blend of pellets was assayed to the counts produced during the calibration experiment was the factor used to normalize the blend data to the calibration experiment.

Table 2 summarizes the calibration curve data for each composition of fuel pellet. Also given are typical values of the percentage standard deviation in the w/o of  $^{235}\text{U}$  from the calibration curve. The method used to obtain the calibration curve error is derived in the following section.

#### B. Calibration Curve Error Analysis

The model chosen to calibrate the counts/gram of pellet, as determined by the delayed neutron pellet assay gage, to the w/o total uranium, as determined by chemistry, is

$$y_{ij} = a_1 + a_2(x_i + \epsilon_{x_i}) + a_3(x_i + \epsilon_{x_i})^2 + \epsilon_{y_{ij}}$$

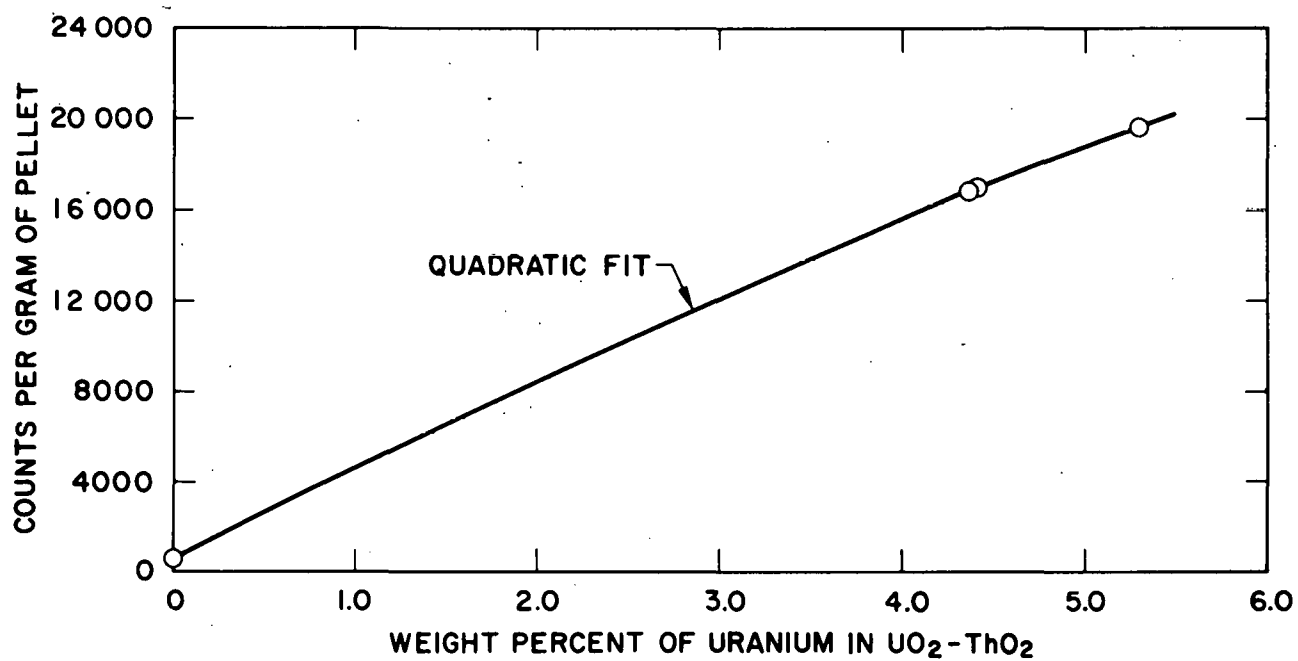


Figure 8. Seed Calibration Curve

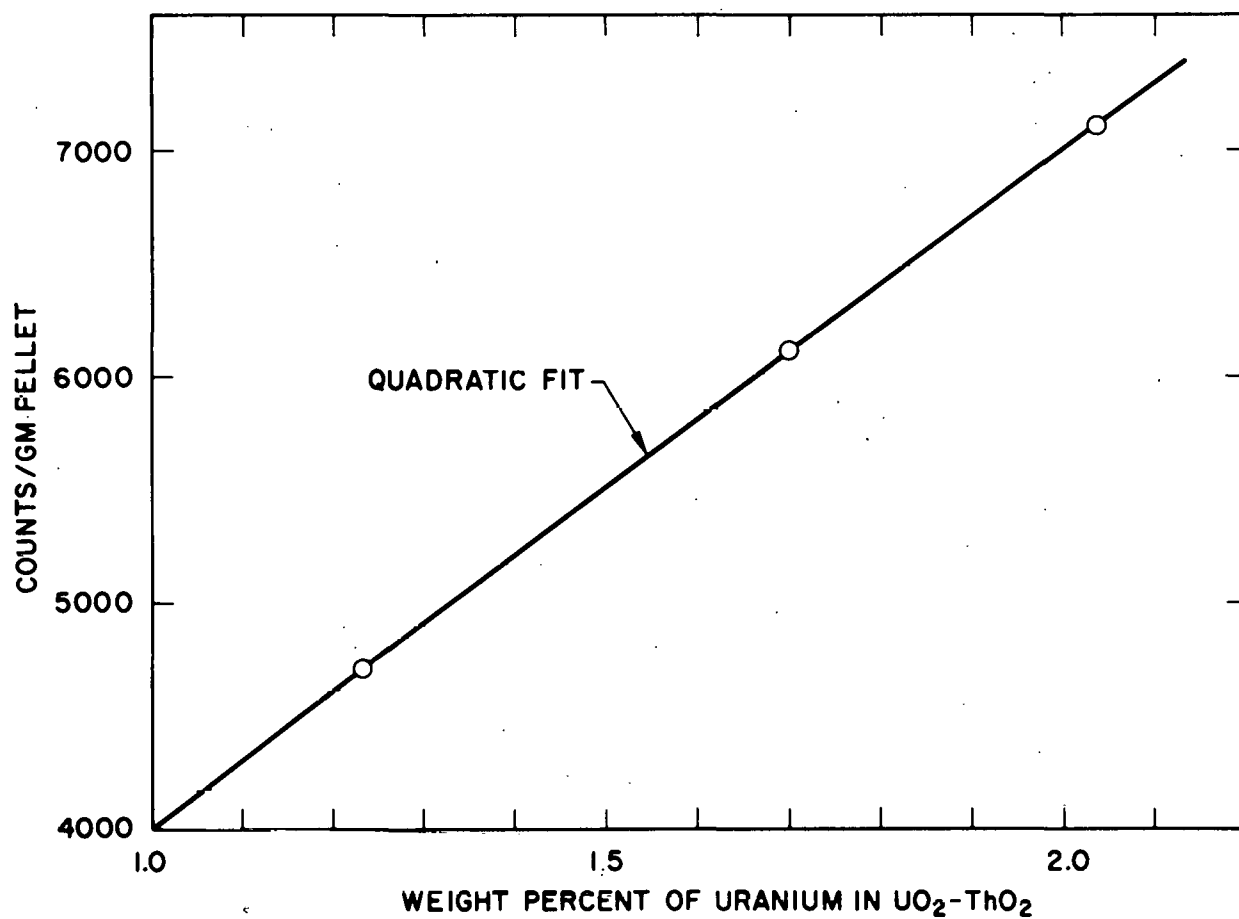


Figure 9. Blanket Calibration Curve

TABLE 2. SUMMARY OF CALIBRATION CURVE DATA

Composition	Fitted Quadratic Coefficients*			Typical $\sigma_{cal}$ (%)
	$a_1$	$a_2$	$a_3$	
Low zone seed	242.646	4361.77	-135.256	0.0390
High zone seed	274.471	4843.55	-156.651	0.0468
Low standard blanket	382.072	6083.63	-294.987	0.0364
Medium standard blanket	622.189	11147.30	-828.603	0.0288
High standard blanket	857.995	3219.26	-74.038	0.0347
Low power flattening blanket	249.995	3551.70	-184.860	0.0312
Medium power flattening blanket	1023.640	2802.78	-8.140	0.0329
High power flattening blanket	-210.262	4047.40	-316.451	0.0312

\* $y = a_1 + a_2x + a_3x^2$  where  $x$  is the w/o of  $^{235}\text{U}$  in the pellet and  $y$  is the counts per gram of pellet in the prescribed counting period.

where

$y_{ij}$  = the  $j$ th counts/gram reading for the  $i$ th pellet,

$x_i$  = is the w/o of  $^{235}\text{U}$  in the  $i$ th  $^{235}\text{UO}_2$ - $\text{ThO}_2$  pellet, obtained from chemistry,

$a_1, a_2, a_3$  = constants to be determined,

$\epsilon_{y_{ij}}$  = the error involved in the observation of  $y_{ij}$ , and

$\epsilon_{x_i}$  = the error in the w/o value obtained by chemistry.

The counts per gram were corrected for background counts. Also outliers were replaced by a boundary value using the method discussed in the Appendix.

The equation may be rewritten as

$$y_{ij} = a_1 + a_2x_i + a_3x_i^2 + \epsilon_{o_{ij}}$$

where the total error contribution is

$$\epsilon_{o_{ij}} = \epsilon_{y_{ij}} + (a_2 + 2a_3x_i) \epsilon_{x_i},$$

assuming  $\epsilon_{x_i}^2$  is sufficiently small that it may be ignored, i.e.,  $|\epsilon_{x_i}| \ll x_i$ , and  $x_i$  is now the fixed observed value.

A weighted least squares analysis using a modified version of Reference 5 was made to establish the calibration curve. The squares of the differences  $y_{ij} - a_1 - a_2x_i - a_3x_i^2$  are weighted by the inverse of the variance of  $\epsilon_{o_{ij}}$ . From above,

$$\text{Var}(\epsilon_{o_{ij}}) = \sigma_{y_{ij}}^2 + (a_2 + 2a_3x_i)^2 \sigma_{c_i}^2$$

where

$$\sigma_{y_{ij}}^2 = \text{Var}(\epsilon_{y_{ij}})$$

and

$$\sigma_{c_i}^2 = \text{Var}(\epsilon_{x_i}).$$

The counts/gram value  $y_{ij}$  is the ratio of the total counts  $TC_{ij}$  for the  $j$ th reading of the  $i$ th pellet, divided by the weight  $G_i$  of the  $i$ th pellet in grams. Since counts have a Poisson distribution, the variance of the distribution of  $TC_{ij}$  is the mean of the distribution and is estimated by  $TC_{ij}$  itself. Assuming that the fractional error in pellet weight  $G_i$  is negligible compared to the fractional error in  $TC_{ij}$ , the estimated variance of  $y_{ij}$  is

$$\sigma_{y_{ij}}^2 = \frac{TC_{ij}}{G_i^2} = \frac{y_{ij}}{G_i}.$$

Hence, the estimate of the error variance  $\text{Var}(\epsilon_{o_{ij}})$  for the observations  $y_{ij}$  and  $x_i$  is

$$\hat{\sigma}_{o_{ij}}^2 = \frac{y_{ij}}{G_i} + (a_2 + 2a_3x_i)^2 \sigma_{c_i}^2$$

where  $\sigma_{c_i}^2 = (0.0025)^2 x_i^2$  and 0.0025 is a conservative estimate of the fractional error from chemistry.



The weighted least squares procedure is to minimize

$$S(y) = \sum_i \sum_j \left[ \frac{y_{ij} - a_1 - a_2 x_i - a_3 x_i^2}{\hat{\sigma}_{o_{ij}}} \right]^2$$

This gives the fitted equation

$$\hat{y}_i = \hat{a}_1 + \hat{a}_2 x_i + \hat{a}_3 x_i^2$$

where  $a_1$ ,  $a_2$ , and  $a_3$  are functions of all the  $x_i$  and  $y_{ij}$  from the calibration blend. The  $a$ 's are random variables, since they will vary from one application of the calibration experiment to another. The least squares analysis yields an estimated residual variance  $\hat{\sigma}_o^2$  and a covariance matrix  $B = \{b_{lk}\}$  for the fitted parameters  $\hat{a}_1$ ,  $\hat{a}_2$ , and  $\hat{a}_3$ . The variance of fitted value  $\hat{y}_i$  at any arbitrary fixed point  $x_i$  (not necessarily one of the values from the calibration blends) is given by

$$\hat{\sigma}_{\hat{y}_i}^2 = \begin{pmatrix} 1 & x_i & x_i^2 \end{pmatrix} B \begin{pmatrix} 1 \\ x_i \\ x_i^2 \end{pmatrix} = b_{11} + 2b_{12}x_i + (2b_{13} + b_{22})x_i^2 + 2b_{23}x_i^3 + b_{33}x_i^4$$

The arbitrary point  $x_i$  will be used below to represent any one of the 20 w/o values of the pellets to be assayed from a production blend. The variance  $\hat{\sigma}_{\hat{y}_i}^2$  is the variance due to the uncertainty in the calibration curve itself. To obtain the uncertainty of the calibration curve for  $x_i$  give some  $y$  value, the equation

$$y_i = \hat{a}_1 + \hat{a}_2 x_i + \hat{a}_3 x_i^2 \pm \hat{\sigma}_{\hat{y}_i}$$

was solved for lower and upper bounds on  $x_i$ :  $x_\ell < x_i < x_u$ . The standard deviation of  $x_i$  due to the uncertainty in the calibration curve was taken to be

$$\hat{\sigma}_{\text{cal } x_i} = \max[x_u - x_i, x_i - x_\ell]$$

The relative standard deviation is then

$$(\hat{\sigma}\%)_{\text{cal } x_i} = \frac{100}{x_i} \hat{\sigma}_{\text{cal } x_i}$$

Thus, for any blend, the counts per gram would be measured for each of the 20 pellets being assayed. The calibration curve would then give the estimate of w/o of  $^T\text{U}$ ,  $x_i$ , and the uncertainty in the curve would cause an uncertainty  $(\hat{\sigma}\%)_{\text{cal } x_i}$  in  $x_i$ . In practice the 20 values of  $(\hat{\sigma}\%)_{\text{cal } x_i}$  for a given blend were almost the same. The maximum of the 20 values was used as the blend calibration error. This calibration curve error is used in Section VI.B, where the error on the gage estimate of the blend weight percent is derived.

## VI. GAGE ESTIMATE OF BLEND WEIGHT PERCENT AND ERROR

### A. Gage Estimate of Blend Weight Percent

For each composition, a separate calibration curve was established, as discussed in Section V. Twenty randomly selected pellets from each blend were assayed with the gage along with the calibration retainer samples and a high count rate standard. Each pellet was assayed three times and a test was applied to determine if an outlier existed in the data. The count data were background corrected, adjusted for counting rate losses, drift corrected using the high count rate standard data, normalized to the calibration blend using the retainer pellet data, and then scanned for outliers. The counts per gram of pellet were adjusted for individual pellet differences in length, diameter, density, and isotopic content between the unknown and calibration blends.

In Sections VI and VII upper case letters will be used for the observed values for production blends determined with the gage. Thus, let  $\bar{Y}_i$  = counts per gram of pellet measured with the gage for the  $i$ th pellet of a production (as opposed to calibration) blend which was assayed. The bar indicates an average of three measurements for the  $i$ th pellet. Let  $X_i$  = w/o of  $^T\text{U}$  for the  $i$ th pellet of a production blend determined from  $\bar{Y}_i$  and the appropriate calibration curve.

To obtain the weight percent of  $^T\text{U}$  for each pellet, values of the fitted calibration curve coefficients were used as follows:

$$X_i = \frac{-\hat{a}_2 + \sqrt{\hat{a}_2^2 - 4(\hat{a}_1 - \bar{Y}_i)\hat{a}_3}}{2\hat{a}_3}$$

## B. Gage Assay Error

The gage error in the weight percent of  $^{235}\text{U}$  for a blend consists of the calibration error and the error due to the gage and pellet variables. This was derived as follows:

Let

$$\bar{Y}_i = A\bar{C}_i$$

where

$\bar{C}_i$  = counts per gram for pellet  $i$  before adjustments and  $A$  is an adjustment for isotopic and normalization correction.

The other corrections listed in Section VI.A. are incorporated directly into the count data.

For  $k$  assays of pellet  $i$ , the variance in the average counts per gram of each pellet is

$$(\sigma\%)_{\bar{Y}_i}^2 = \frac{(\sigma\%)_o^2}{k} + (\sigma\%)_p^2 + (\sigma\%)_A^2$$

where  $(\sigma\%)_o$  represents the within-pellet standard deviation resulting from repeated counting of each pellet within a blend and  $(\sigma\%)_p$  represents the pellet-to-pellet standard deviation, both relative to  $\bar{C}_i$ . Both  $(\sigma\%)_o^2$  and  $(\sigma\%)_p^2$  are estimated from the counts per gram data using standard analysis of variance techniques. Also,

$$(\sigma\%)_A^2 = (\sigma\%)_I^2 + (\sigma\%)_N^2$$

where

$\sigma_I$  = standard deviation of the isotopic correction

$\sigma_N$  = standard deviation of the normalization correction.

For  $n_g$  pellets per blend assayed by the gage, the variance in the blend average counts per gram is

$$(\sigma\%)_{\bar{Y}}^2 = \frac{(\sigma\%)_{\bar{C}}^2}{n_g} + (\sigma\%)_A^2$$

where

$$(\sigma\%)^2_{\bar{c}} = \frac{(\sigma\%)^2_o}{k} + (\sigma\%)^2_p.$$

The error in the standard deviation of the blend average weight percent  $\bar{X}$ , assuming a perfect calibration curve, is obtained by dividing  $\sigma_{\bar{Y}}$  by the slope of the calibration curve at the weight percent of interest; i.e.,

$$b = \left. \frac{dy}{dx} \right|_{x = \bar{X}}$$

and

$$(\sigma\%)^2_{\bar{X}} = \left[ 100 \frac{\sigma_{\bar{X}}}{\bar{X}} \right]^2 = \left[ \frac{100}{\bar{X}} \frac{\sigma_{\bar{Y}}}{b} \right]^2 = \left[ \frac{\bar{Y}}{\bar{X}} \frac{(\sigma\%)_{\bar{Y}}}{b} \right]^2$$

Finally, the total relative variance of the gage value of the blend weight percent is

$$\text{Var}(\bar{X})_{\%} = (\hat{\sigma}\%)^2_{\text{cal}} + (\sigma\%)^2_{\bar{X}}.$$

## VII. DETERMINATION OF BLEND WEIGHT PERCENT AND UNCERTAINTY

### A. Summary of Methods Used

From each production blend, 20 pellets were assayed by the gage, and from these 20 pellets, 5 were analyzed by chemistry. The blend mean weight percent was based upon a maximum likelihood estimate of the combined gage and chemistry results. The expression for the mean value is given in Equation (1) and its variance in Equation (2). The derivation of these expressions and definition of terms is given in Section VII.B.

$$\hat{\mu} = \frac{\bar{X}\bar{V}_{\bar{c}} + \bar{c}\bar{V}_{\bar{X}}}{\bar{V}_{\bar{X}} + \bar{V}_{\bar{c}}} \quad (1)$$

where

$$\bar{V}_{\bar{X}} = \text{Var}(\bar{X}) - \text{Cov}(\bar{X}, \bar{c})$$

$$\bar{V}_{\bar{c}} = \text{Var}(\bar{c}) - \text{Cov}(\bar{X}, \bar{c})$$

$\bar{V}_{\bar{X}}$  = the variance of the average of  $n_g$  pellets assayed by the gage per blend

$\text{Var } \bar{c}$  = the variance of the average of  $n_c$  pellets analyzed by chemistry per blend

$\text{Cov}(\bar{X}, \bar{c})$  = the covariance between  $\bar{X}$  and  $\bar{c}$  as defined in Section VII.B

$\bar{X}$  = average weight percent for samples analyzed by the gage

$\bar{c}$  = average weight percent for samples analyzed by chemistry.

$$\text{Var}(\hat{\mu}) = \frac{\frac{V_c^2}{c} V(\bar{X}) + \frac{V_X^2}{\bar{X}} \text{Var}(\bar{c}) + 2V_c V_X \text{Cov}(\bar{X}, \bar{c})}{(V_c + V_X)^2}$$

The five pellets that were analyzed by chemistry and assayed by the gage were used as a control to ensure that the two methods continued to produce consistent results. Two tests were performed on the pellets assayed by both methods. The first test required that the average difference between chemistry results and the gage results be less than  $3 \sigma_{\bar{d}}$ , where  $\bar{d}$  and the standard deviation  $\sigma_{\bar{d}}$  are given by

$$\bar{d} = \bar{X} - \bar{c}$$

$$\sigma_{\bar{d}} = \sqrt{V_c + V_X}$$

where  $V_c$  and  $V_X$  are defined as above, and for this case  $n_c = n_g = 5$ .

Assuming normality, this test could be expected to fail about three times out of a thousand due solely to chance. If the test were not passed, then chemistry and gage data were scrutinized to see if any outlying data could be present. The procedure used to detect the presence of an outlier is described in the Appendix. Briefly, if an outlier was found in either set of data, it was replaced by the value calculated per the procedure and the test performed again. If the test was not passed, then chemistry would perform analysis on two additional pellets from the blend which were also processed through the gage. A new average difference was determined including the additional data and the test performed again. If the test was still not passed, then the gage calibration was checked using all the retainer samples left for use as standards for each composition. If the calibration curve was correct, then the failure to meet this test requirement could indicate a defective pellet blend. The blend could have excessive inhomogeneity, contamination, or an improper isotopic composition ascribed to it. There were, however, no defective pellet blends found as a result of the assay of pellets by the gage and by chemistry.

The second test was designed to detect a bias which may have developed between the gage and chemistry. This test was conducted by observing the sign of the average difference between the gage and chemistry. Keeping approximately the same failure probability of the test (3 out of 1,000) implied that the test would fail if nine successive blends had the same algebraic sign of the average difference between the gage and chemistry. If this test was failed, then a bias was applied to the gage results for the preceding nine blends. This bias continued to be applied until the test was failed again or until completion of the assay of all blends from a given composition. This bias was defined as the average of the differences between gage and chemistry for the nine blends. The gage results required bias correction six times throughout the entire pellet production program which covered over 887 blends.

#### B. The Chemistry Average Variance and Gage-Chemistry Covariance

To establish a combined maximum likelihood estimate for the mean blend w/o  $T_U$  from data from both the gage and chemistry, the variance for the average chemistry w/o value and the covariance between the gage and chemistry values are required.

Let

$$c_i = \mu + \epsilon_{c_i} + \epsilon_{p_i}$$

where

$c_i$  = the chemistry value in w/o  $T_U$  for the  $i$ th pellet from the blend,

$i = 1, 2, \dots, n_c$

$\mu$  = the true mean pellet w/o  $U$  for the blend

$\epsilon_{c_i}$  = the error from the chemical analysis

$\epsilon_{p_i}$  = the error due to pellet-to-pellet variation (in w/o).

Then

$$\text{Var}(c_i)_{\%} = (\sigma\%)_c^2 + (\sigma\%)_p^2$$

where

$(\sigma\%)_c^2$  = the chemistry variance relative to  $c_i$ , and

$(\sigma\%)_p^2$  = the pellet-to-pellet variance relative to the pellet w/o  $T_U$  but as estimated from the replicated gage data.

Since each pellet is chemically analyzed only once, the relative variance for the chemistry average is approximately

$$\text{Var}(\bar{c})_{\%} = \text{Var}\left[\frac{1}{n_c} \sum c_i\right]_{\%} = \left((\sigma\%)_c^2 + (\sigma\%)_p^2\right) / n_c$$

Since  $n_c$  pellets are analyzed by both gage and chemistry, the only common source of error in  $X_i$  and  $c_i$  is the pellet error. Thus, the relative covariance is

$$\text{Cov}(X_i, c_i)_{\%} = (\sigma\%)_p^2, \quad i = 1, 2, \dots, n_c$$

Averaging over  $n_g$  pellets for the gage and  $n_c$  pellets for chemistry, which were also analyzed by the gage, the relative covariance of the averages can be derived as follows:

$$\begin{aligned} \text{Cov}(\bar{X}, \bar{c})_{\%} &= \text{Cov}\left[\frac{1}{n_g} \sum_{i=1}^{n_g} X_i, \frac{1}{n_c} \sum_{i=1}^{n_c} c_i\right]_{\%} \\ &= \frac{1}{n_g n_c} \sum_{i=1}^{n_c} \text{Cov}(X_i, c_i)_{\%} \\ &= \frac{1}{n_g} (\sigma\%)_p^2 \end{aligned}$$

Two estimates of the mean blend w/o  $^{235}\text{U}$  in  $^{235}\text{UO}_2\text{-ThO}_2$  are available:

$\bar{X}$  = the average of the  $n_g$  calibrated readings from the pellet assay gage, and

$\bar{c}$  = the average of the chemical analysis of  $n_c$  pellets from the same blend.

Assuming  $\bar{X}$  and  $\bar{c}$  are normally distributed, both with mean  $\mu$  and covariance and variances given above, a maximum likelihood estimate for  $\mu$  can be obtained by minimizing the sum of squares functions:

$$\begin{aligned} &[(\bar{X} - \mu)(\bar{c} - \mu)] \Sigma^{-1} \begin{bmatrix} \bar{X} - \mu \\ \bar{c} - \mu \end{bmatrix} \\ &= \frac{[\text{Var}(\bar{c})](\bar{X} - \mu)^2 - 2[\text{Cov}(\bar{X}, \bar{c})](\bar{X} - \mu)(\bar{c} - \mu) + [\text{Var}(\bar{X})](\bar{c} - \mu)^2}{[\text{Var}(\bar{X})][\text{Var}(\bar{c})] - [\text{Cov}(\bar{X}, \bar{c})]^2} \end{aligned}$$

where

$$\Sigma = \begin{bmatrix} \text{Var}(\bar{X}) & \text{Cov}(\bar{X}, \bar{c}) \\ \text{Cov}(\bar{X}, \bar{c}) & \text{Var}(\bar{c}) \end{bmatrix}$$

Differentiating with respect to  $\mu$ , setting the resulting equation equal to zero, and solving for  $\mu$  gives:

$$\begin{aligned} \hat{\mu} &= \frac{[\text{Var}(\bar{c}) - \text{Cov}(\bar{X}, \bar{c})]\bar{X} + [\text{Var}(\bar{X}) - \text{Cov}(\bar{X}, \bar{c})]\bar{c}}{[\text{Var}(\bar{c}) - \text{Cov}(\bar{X}, \bar{c})] + [\text{Var}(\bar{X}) - \text{Cov}(\bar{X}, \bar{c})]} \\ &= \frac{\bar{X} V_{\bar{c}} + \bar{c} V_{\bar{X}}}{V_{\bar{X}} + V_{\bar{c}}} \end{aligned}$$

where

$$\begin{aligned} V_{\bar{X}} &= \text{Var}(\bar{X}) - \text{Cov}(\bar{X}, \bar{c}) \\ V_{\bar{c}} &= \text{Var}(\bar{c}) - \text{Cov}(\bar{X}, \bar{c}). \end{aligned}$$

It can be easily shown that  $\hat{\mu}$  is an unbiased estimator for  $\mu$ , since  $E(X) = E(c) = \mu$ , and that

$$\text{Var}(\hat{\mu}) = \frac{\frac{V_{\bar{c}}^2}{\bar{c}} \text{Var}(\bar{X}) + \frac{V_{\bar{X}}^2}{\bar{X}} \text{Var}(\bar{c}) + 2 \frac{V_{\bar{X}}}{\bar{X}} \frac{V_{\bar{c}}}{\bar{c}} \text{Cov}(\bar{X}, \bar{c})}{(V_{\bar{X}} + V_{\bar{c}})^2}$$

### C. Results

For each blend of each composition the gage, chemistry, and combined results were stored in a computer file. These data plus the weight of the fuel pellets in each fuel rod produced from that blend were used to obtain the rod uranium loading and its error. For each rod, it was required that the gage and chemistry data combined with the weighing uncertainty and the pellet-to-pellet variability be such that the fuel rod loading could be determined with at least 0.25% precision at the 95% confidence level. All of the LWBR fuel rods met this requirement.

Tables 3 and 4 present a summary of the data for each composition. These tables present only the average parameters obtained from the blends of each composition since the individual blend data are too extensive to be reported here. In Table 3 the number of blends per composition, the arithmetic averages of the gage and chemistry w/o's, the combined uranium weight percent ( $\hat{\mu}$ ) and its standard deviation, and the pellet-to-pellet variability are given.



TABLE 3. SUMMARY OF BLEND ASSAY DATA\*

<u>Composition</u>	<u>No. Blends</u>	<u>Gage Average w/o <math>T_U</math> (20 pellets/blend)</u>	<u>Chemistry Average w/o <math>T_U</math> (5 pellets/blend)</u>	<u><math>\sigma_p</math> (%)</u>	<u><math>\hat{\mu}</math> w/o <math>T_U</math></u>	<u><math>\sigma(\hat{\mu})</math> (%)</u>
Low seed	148	4.4082	4.4081	0.098	4.4081	0.092
High seed	203	5.2864	5.2878	0.100	5.2871	0.087
Low standard blanket	39	1.2338	1.2339	0.107	1.2338	0.087
Medium standard blanket	191	1.6959	1.6948	0.084	1.6955	0.077
High standard blanket	73	2.0386	2.0378	0.072	2.0382	0.078
Low power flattening blanket	20	1.6804	1.6812	0.086	1.6807	0.077
Medium power flattening blanket	20	2.0433	2.0453	0.115	2.0442	0.083
High power flattening blanket	114	2.7909	2.7907	0.100	2.7908	0.087

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\*Data represent blends used in the LWBR core.

TABLE 4. COMPARISON OF THE GAGE AND CHEMISTRY RESULTS\*

<u>Composition</u>	<u>No. Blends</u>	<u>Gage Average w/o <math>T_U</math> (5 pellets/blend)</u>	<u>Chemistry Average w/o <math>T_U</math> (5 pellets/blend)</u>	<u>Percent Difference (Gage-Chemistry)</u>
Low seed	148	4.4087 $\pm$ 0.0010	4.4081 $\pm$ 0.0009	0.014
High seed	203	5.2869 $\pm$ 0.0008	5.2878 $\pm$ 0.0008	-0.017
Low standard blanket	39	1.2335 $\pm$ 0.0005	1.2339 $\pm$ 0.0005	-0.032
Medium standard blanket	191	1.6959 $\pm$ 0.0003	1.6948 $\pm$ 0.0003	0.065
High standard blanket	73	2.0387 $\pm$ 0.0005	2.0378 $\pm$ 0.0006	0.044
Low power flattening blanket	20	1.6807 $\pm$ 0.0008	1.6812 $\pm$ 0.0009	-0.030
Medium power flattening blanket	20	2.0436 $\pm$ 0.0010	2.0453 $\pm$ 0.0011	-0.083
High power flattening blanket	114	2.7904 $\pm$ 0.0005	2.7907 $\pm$ 0.0007	-0.011

\*Data represent blends used in the LWBR core.

Table 4 summarizes the composition averaged values of the weight percent of total uranium predicted by the assay gage for the same five pellets per blend that were also analyzed destructively by chemistry. This table lists the average gage and chemistry values, their percent difference, and their uncertainties.

Figure 10 presents a typical distribution of the blend pellet-to-pellet variability. This figure shows the results obtained for the medium standard blanket pellets.

The uncertainty in the blend estimate of the weight percent of total uranium when combined with the uncertainties in the blend isotopic composition, pellet stack weighing, and pellet-to-pellet variability produce a rod loading uncertainty of better than 0.25% precision at the 95% confidence level. The use of the blend estimate of the w/o  $^{235}\text{U}$  to obtain the fuel rod loading uncertainty and the fuel composition loading uncertainty is presented in Reference 6.

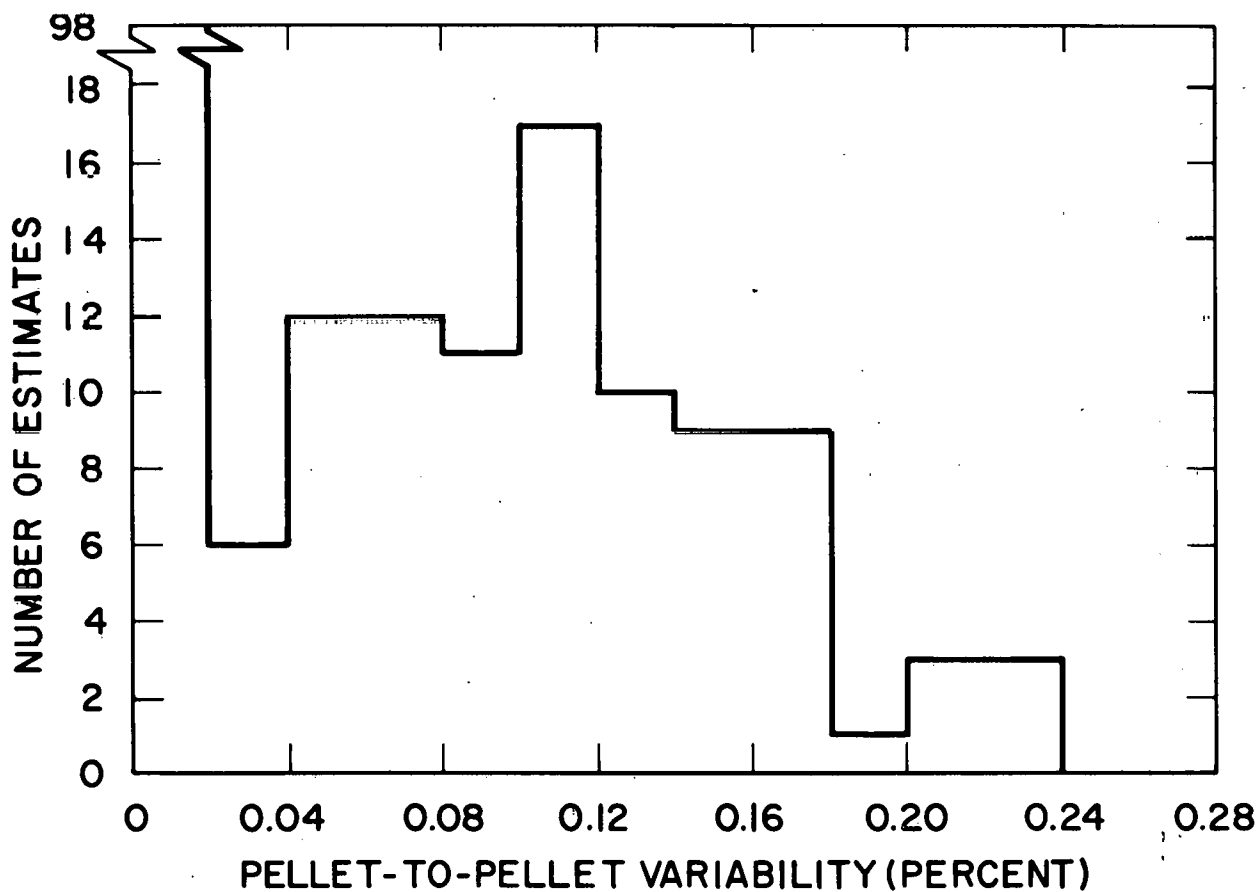


Figure 10. Medium Standard Blanket Distribution of the Pellet-to-Pellet Variability

## VIII. CONCLUSION

The delayed neutron pellet assay gage, when calibrated against chemistry, can be used to measure with high precision the weight percent and macroscopic homogeneity of total uranium for fuel pellets of similar composition and physical properties. For each blend of fuel pellets used in the manufacture of the LWBR core, a maximum likelihood estimate of the blend mean weight percent of total uranium was determined. This estimate was based upon the chemical assay of five pellets per blend and by the delayed neutron assay of 20 pellets per blend. Use of the delayed neutron pellet assay gage reduced the amount of chemistry that would have been required to achieve the same fuel rod loading precision by a factor of two.

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## APPENDIX

### TREATMENT OF OUTLIERS

A variation of the semiwinsorization treatment of outliers, Reference 7, was used in conjunction with the analysis of the delayed neutron pellet assay gage data. In this scheme, the detected outlier was replaced by a boundary value. Outliers were detected at the 1% level and were adjusted to the 5% level. The data were first scanned for those values for the counts per gram of pellet such that

$$T_i = \frac{X_{ie} - \bar{X}_i}{\hat{\sigma}_{w(20)}} > T_{K,20(K-1),0.05} \quad (1)$$

where

$\bar{X}_i$  = the average counts/gm for each pellet for all passes

$X_{ie}$  = the extreme individual value of counts/gm contained in  $\bar{X}_i$

$\hat{\sigma}_{w(20)}$  = the pooled estimate of the standard deviation for within-pellet variation for 20 pellets

$T_{K,20(K-1),0.05}$  = a critical value for detecting one outlier from K observations per pellet at the 5% level.

Having flagged these values at the 5% level, the above test is again applied to the largest such value using a 1% level and  $\hat{\sigma}_w$  from the remaining 19 pellets. If no values were flagged at the 5% level, then the processing of the data may continue with no changes. If one or more values were flagged at the 5% level, the largest such value is tested as being an outlier at the 1% level. If this value turns out not to be an outlier, then no action is taken and the data are processed normally. If the observation is an outlier, then the outlier is replaced by a boundary value as calculated below. The test and replacement of an outlier are shown below:

$$T_i = \frac{X_{ie} - \bar{X}_i}{\hat{\sigma}_{w(19)}}$$

if  $T_i \leq T_{K,v,0.01}$ , then the data contain no outliers;

if  $T_i > T_{K,v,0.01}$ , then  $X_{ie}$  is replaced by the boundary value  $X'_{ie}$  given by

$$X'_{ie} = \bar{X}_{i'(K-1)} \pm T_{K,v,0.05} \hat{\sigma}_{w(19)} \quad (2)$$

where

$\bar{X}_{i,(K-1)}$  = average counts per gram for the pellet in which the outlier was detected with the outlier deleted from the average

$T_{K,v,0.05}$  = critical value for K observations per each pellet, for v degrees of freedom, at the 0.05 level of significance

$v = 19(K-1)$  if there were twenty pellets originally and one pellet contained an outlier

$\hat{\sigma}_{w(19)}$  = the pooled estimate of the within-pellet variation for the remaining 19 pellets; i.e., the pellet in which the outlier was detected is excluded.

After the outlier has been replaced by the boundary value, the average counts per gram of pellet are recomputed for the pellet in which the outlier occurred, and for all the pellets a new value of  $\hat{\sigma}_{w(20)}$  is computed. Using these new values, the data are again screened for outliers just as before. This screening and testing procedure is performed only twice, since repeated application of the procedure could lead to an artificially low value of  $\hat{\sigma}_w$ . If a true outlier does not exist in the data, the probability of detecting more than one false outlier from 20 pellets is less than 0.02.