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Test and Evaluation Results of the ^{252}Cf Shuffler at the Savannah River Plant

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TEST AND EVALUATION RESULTS OF THE ^{252}Cf SHUFFLER AT THE SAVANNAH RIVER PLANT

by

T. W. Crane

ABSTRACT

The ^{252}Cf Shuffler, a nondestructive assay instrument employing californium neutron source irradiation and delayed-neutron counting, was developed at Los Alamos National Laboratory for measuring ^{235}U content of scrap and waste items generated at the Savannah River Plant (SRP) reactor fuel fabrication facility. The scrap and waste items include high-purity uranium-aluminum alloy ingots as well as pieces of castings, saw and lathe chips from machining operations, low-purity items such as oxides of uranium or uranium intermixed with flux materials found in recovery operations, and materials not recoverable at SRP such as floor sweepings or residues from the uranium scrap recovery operation. The uranium contains about 60% ^{235}U with the remaining isotopes being ^{236}U, ^{238}U, and ^{234}U in descending order. The test and evaluation at SRP concluded that the accuracy, safety, reliability, and ease of use made the ^{252}Cf Shuffler a suitable instrument for routine use in an industrial, production-oriented plant.

I. INTRODUCTION

A. Test and Evaluation Program

The ^{252}Cf Shuffler built for Savannah River Plant (SRP) is a nondestructive assay (NDA) instrument designed to measure the ^{235}U content of scrap and waste items at the SRP reactor fuel fabrication facility.[1] The program to test and evaluate the Shuffler at SRP is supported by the US Department of Energy (DOE) Office of Safeguards and Security (OSS). Los Alamos National Laboratory (Los Alamos) has been responsible for designing and building the NDA instrument,

while SRP has provided operators, data characterizing their material, a suitable location, and standards. Personnel from Los Alamos have provided training for operators and consultation on locating the Shuffler in the SRP facility and fabricating standards.

At the SRP reactor fuel fabrication facility, uranium and aluminum are alloyed and manufactured into reactor fuel tubes. The process, which includes casting, machining, and extrusion as well as on-site recovery of scrap, is shown schematically in Fig. 1. During the test and evaluation program, the Shuffler has assayed items normally handled at SRP in their standard "scrap can," approximately 30-cm high and 18-cm diam. A summary of the scrap and waste items assayed is found in Table I.[2,3] These scrap and waste items, with the exception of floor sweepings, LX material, and LF flux are recycled at the SRP reactor fuel fabrication facility.

B. Californium Shuffler Measurement Technique

The ^{252}Cf Shuffler uses an NDA technique based upon the delayed-neutron emission rate following irradiation by a strong ^{252}Cf neutron source.[4,5] The active interrogation cycle is illustrated by the two-step sequence shown in Fig. 2. First, the source is transferred to the assay chamber for neutron interrogation of the item being assayed. After interrogation, the source is transferred back to the storage location, and delayed neutrons are counted in the assay chamber. The interrogation and delayed-neutron counting times are 12.2 s each, and the transfer requires about 0.585 s in either direction. Thus, a complete active assay cycle requires 25.6 s, and typically between 6 and 12 cycles are used for an assay. For low-level or high-precision measurements, a maximum of 30 cycles is permitted. A full assay sequence also includes a background neutron measurement (which preceeds the active assay) with the source in the storage position and a weight measurement.

1. Californium Neutron Source Interrogation. Interrogating neutrons originate from spontaneous fissions in the ^{252}Cf neutron source. Of the possible isotopic neutron sources, californium is clearly the best choice because of its high neutron emission rate (2.33×10^{12} neutrons/s/g), reasonable half-life (2.65 years), and availability. The source in Shuffler was ~0.6 mg as of January 1, 1980, yielding about 1.4×10^{9} neutrons/s. Approximately four to five years of service will be provided by the source.

Neutrons from the ^{252}Cf source have a fission energy spectrum with a mean energy of 2.3 MeV. In order to minimize the response from the even uranium

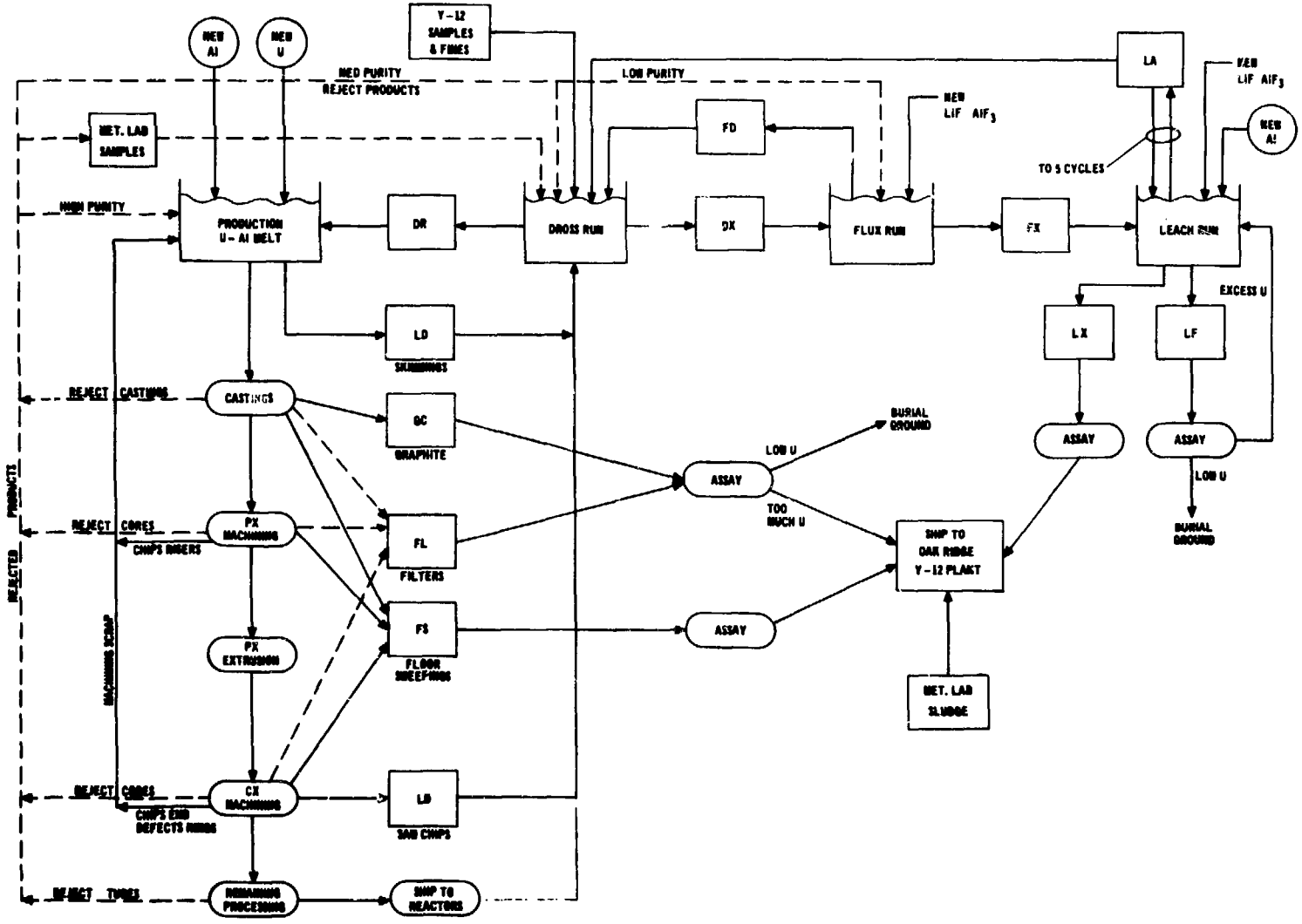


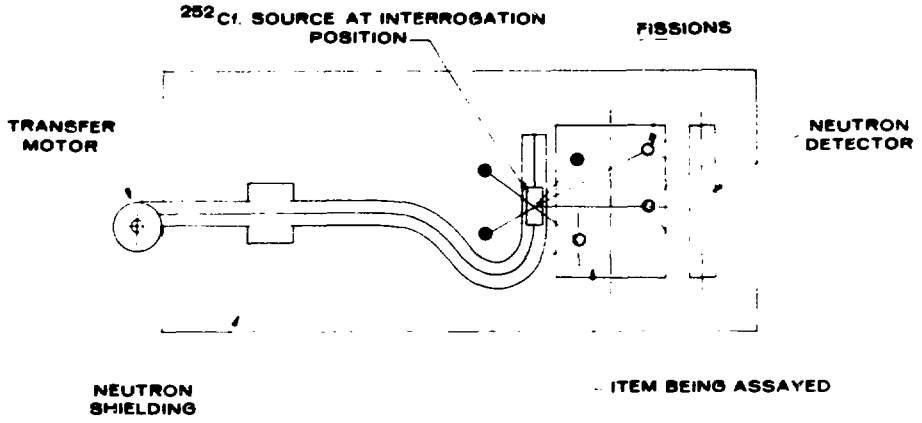
Fig. 1. Material flow diagram for the reactor fuel tube fabrication facility (EG&G Neg. No. 10019R).

TABLE I

SCRAP AND WASTE CATEGORIES MEASURED BY THE ^{252}Cf SHUFFLER AT SRP

Material	Description	Process Location		Specifications	Max.	Min.
DR Ingot	Solid cylindrical alloy ingot, 159-mm diam, 50- to 100-m height stored in scrap cans.	From: Recovered alloy from Dross Run To: Production melt		U-Al (g) ^{235}U (g) Items/day	6 200 985 10	2 342 89 1
DX Ingot	Solid cylindrical ingot containing alloy and uranium and aluminum oxides, 159-mm diam, 50- to 250-mm height. Stored in scrap cans.	From: Dross Run waste To: Flux Run		U-Al (g) ^{235}U (g) Items/day	15 694 2 433 4	2 000 158 1
Skimmings	Dross skimmed from production melts. Larger pieces broken into irregular shapes for storage in scrap cans.	From: Production Melt To: Dross Run		U-Al (g) ^{235}U (g) Items/month	9 894 1 542 57	355 55 33
FD Ingot	Solid cylindrical alloy ingot, 159-mm diam, 100- to 250-mm height, stored in scrap cans.	From: Flux Run recovered U-Al alloy To: Dross Run		U-Al (g) ^{235}U (g) Items/day	14 564 2 257 12	2 064 319 1
FX Ingot	Solid cylindrical ingot, 159-mm diam, 50-250-mm height. Consists of some U-Al alloy with oxides of U and Al dissolved in AlF_3 and LiF fluxes.	From: Flux Run waste To: Leach Run		Items/week	15	1
LA Ingot	Solid alloy ingot 159-mm diam, 100- to 250-mm height. Typically 1-5 weight per cent uranium.	From: Leach Run recovered U-Al alloy To: Dross Run		U-Al (g) ^{235}U (g) Items/day	7 760 112 5	5 000 62 1
LF Ingot	Hollow ingot 240-mm o.d., 305-mm height used from gamma-ray analysis. This material can be broken up or cast in solid ingots for analysis by the Shuffler.	From: Leach Run waste To: Burial ground if less than 0.35 weight per cent ^{235}U.		Items/week	6	1
LX Residues	Irregular chunks of material insoluable in molten flux that remain in the crucible after flux and alloy are poured from a leach run. Contain compounds of C with Al, U, Li, and F.	From: Leach Run To: Oak Ridge		Weight (g) ^{235}U (g) Items/day	14 208 548 2	747 12 1
Loose Dross	Irregular shapes of tube sections and intermediate products from destructive examination samples often mixed with other process scrap alloys. Contained in scrap cans.	From: Metallurgical Lab. To: Dross Run		U-Al (g) ^{235}U (g) Items/day	13 792 2 383 43	355 55 2
Floor Sweepings	Crucible shells, metal spills, pieces of graphite, metal chips dirt, and trash. Stored in scrap cans.	From: Casting and machining area floors To: Oak Ridge		Weight (g) ^{235}U (g) Items/day	7 984 594 8	594 47 1
Risers	Cut from ends of production alloy hollow cylindrical castings, then broken into pieces and stored in scrap cans.	From: Production ingots To: Production melt		U-Al (g) ^{235}U (g) Items/day	8 866 1 566 8	2 130 361 1
Lathe Chips	Lathe chips of U-Al alloy cut from ingots and logs. Stored in scrap cans.	From: Machining room To: Production melt		U-Al (g) ^{235}U (g) Items/day	3 480 600 7	540 419 1
Saw Chips	Saw chips of U-Al alloy cut from logs. Stored in scrap cans.	From: Machining room To: Dross Run		Items/week	3	1

A ^{252}Cf. NEUTRON SOURCE IS USED TO INDUCE FISSIONS IN THE SAMPLE



DELAYED NEUTRONS ARE COUNTED WITH THE SOURCE STORED

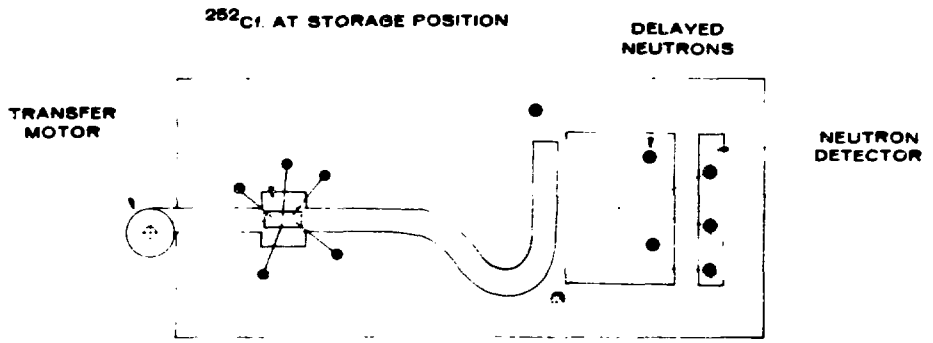


Fig. 2. Californium Shuffler active assay sequence (EG&G Neg. No. 10315).

isotopes in the SRP material (^{234}U, ^{236}U, and ^{238}U), the energy of the source neutrons must be reduced below energies for which the even isotope fission cross section becomes appreciable. On the other hand, if a significant fraction of source neutrons is moderated below the energy where the ^{235}U fission cross section begins to rapidly increase (about 1 keV), then self-shielding will make the assay sensitive to the size, shape, and uranium density of the items being assayed. Extensive neutron transport calculations were used to select the assay chamber materials and geometry that would yield a penetrating neutron spectrum without excessive response from the even uranium isotopes. Figure 3 shows the assay chamber. Nickel and steel are used to provide some moderation, while boron and cadmium are used to absorb low-energy neutrons.

2. Delayed Neutrons From Fission. Delayed neutrons are emitted from fission fragments after one or more beta decays. The half-lives for different delayed-neutron groups range from 0.2 to 55 s and are emitted for about 1.6% of the ^{235}U fissions.[6] Delayed neutrons or even the prompt neutrons cannot be observed during the californium neutron irradiation because the californium

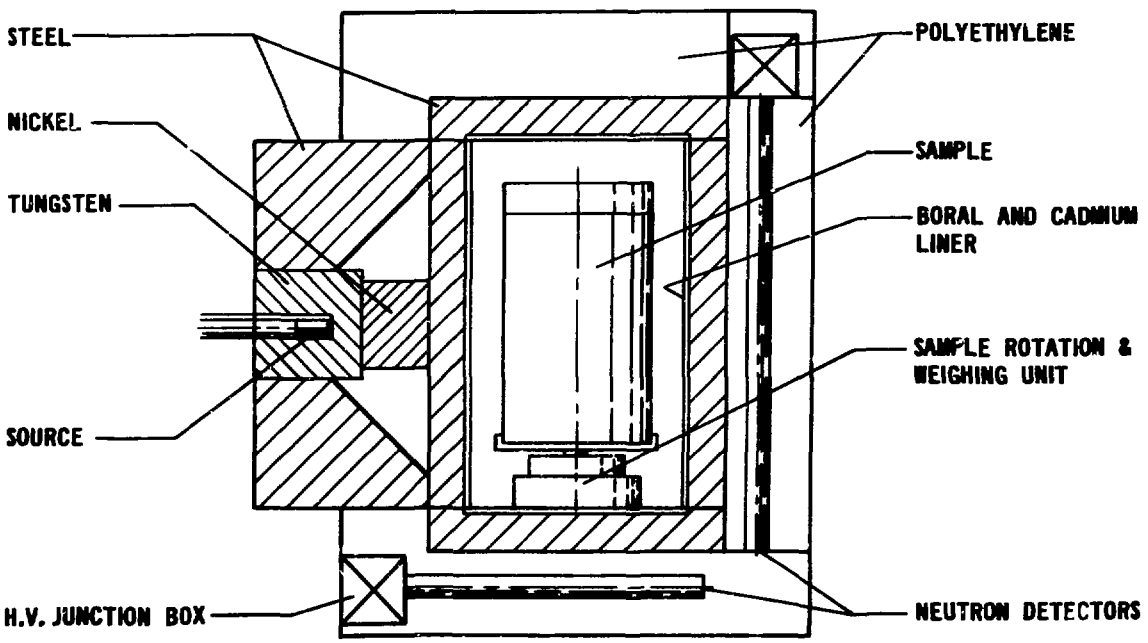


Fig. 3. Californium Shuffler assay chamber (EG&G Neg. No. 10017).

source dominates the detector response. Thus, the assay requires the source to be modulated. Transferring the source to an isolated storage chamber permits delayed-neutron and background counting, and moving the source to the assay chamber accomplishes the neutron irradiation.

II. THE ^{252}Cf SHUFFLER INSTRUMENT

The ^{252}Cf Shuffler NDA system includes the assay unit, electronics rack, and communication terminals shown in Fig. 4. In order to reduce contamination, only the assay unit and a control keyboard were placed in the vault. Figure 5 shows the assay unit installed in the scrap vault. The electronics rack and terminals are located outside the vault as shown in Fig. 6. An operator inside the vault can view the large-format video display through the Plexigas window visible in both Figs. 5 and 6. An intercom system is used for communication between operators inside the vault and supervisory personnel viewing the Shuffler from outside the vault.

A. Hardware Inside the Scrap Vault

Two cubical assemblies and a connecting section compose the assay unit. Each cubical assembly has an edge length of about 1.22 m and a mass of 2700 kg. The internal structure is indicated in Fig. 7. One cube is used for the source storage position and is completely filled with neutron and gamma-ray shielding material. The other cube contains the assay chamber. A small jib crane is used to place items in the chamber.[7,8]

In the base of the assay chamber, load cells are mounted for sample weight measurement. A small motor using a cam-lifting mechanism raises the item off the cells for the "zero" weight measurement and allows sample rotation. The weight is used for alternative item identification and as part of the input to the correction factor based upon the uranium weight percentage in the U-Al alloy.

The neutron detectors surrounding the assay chamber are ^{3}He-filled proportional gas tubes and are used for delayed-neutron counting. The ratio of the counts in the side and bottom banks can be used to determine the approximate fill height of items being assayed.[1,7] Fill height data are used as part of the correction factor estimation. Two small detectors located inside the assay chamber measure the neutron flux from the californium source during irradiation.

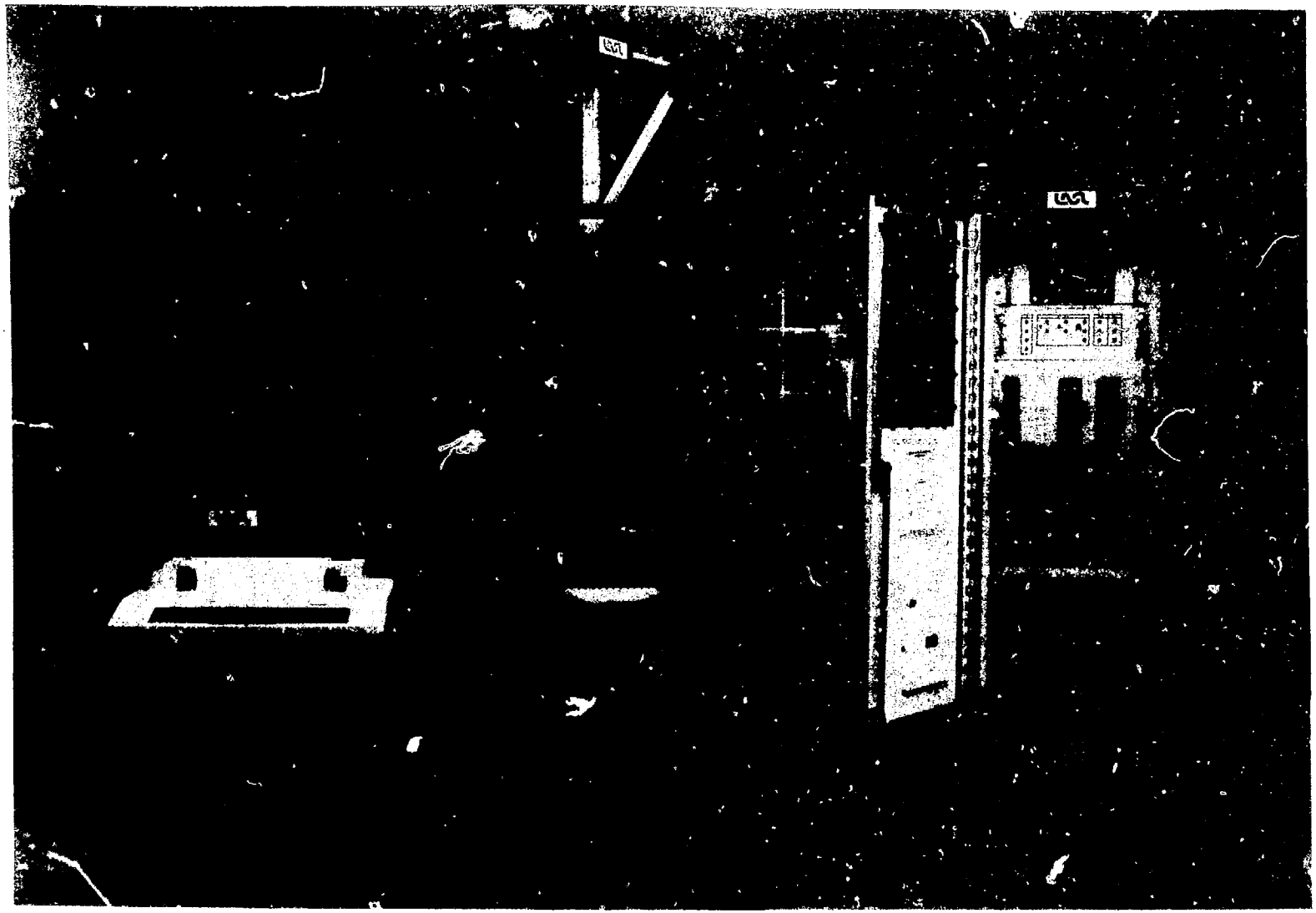


Fig. 4. Complete ^{252}Cf Shuffler assay system after assembly at Los Alamos (Los Alamos Neg. No. CN 78-6496).

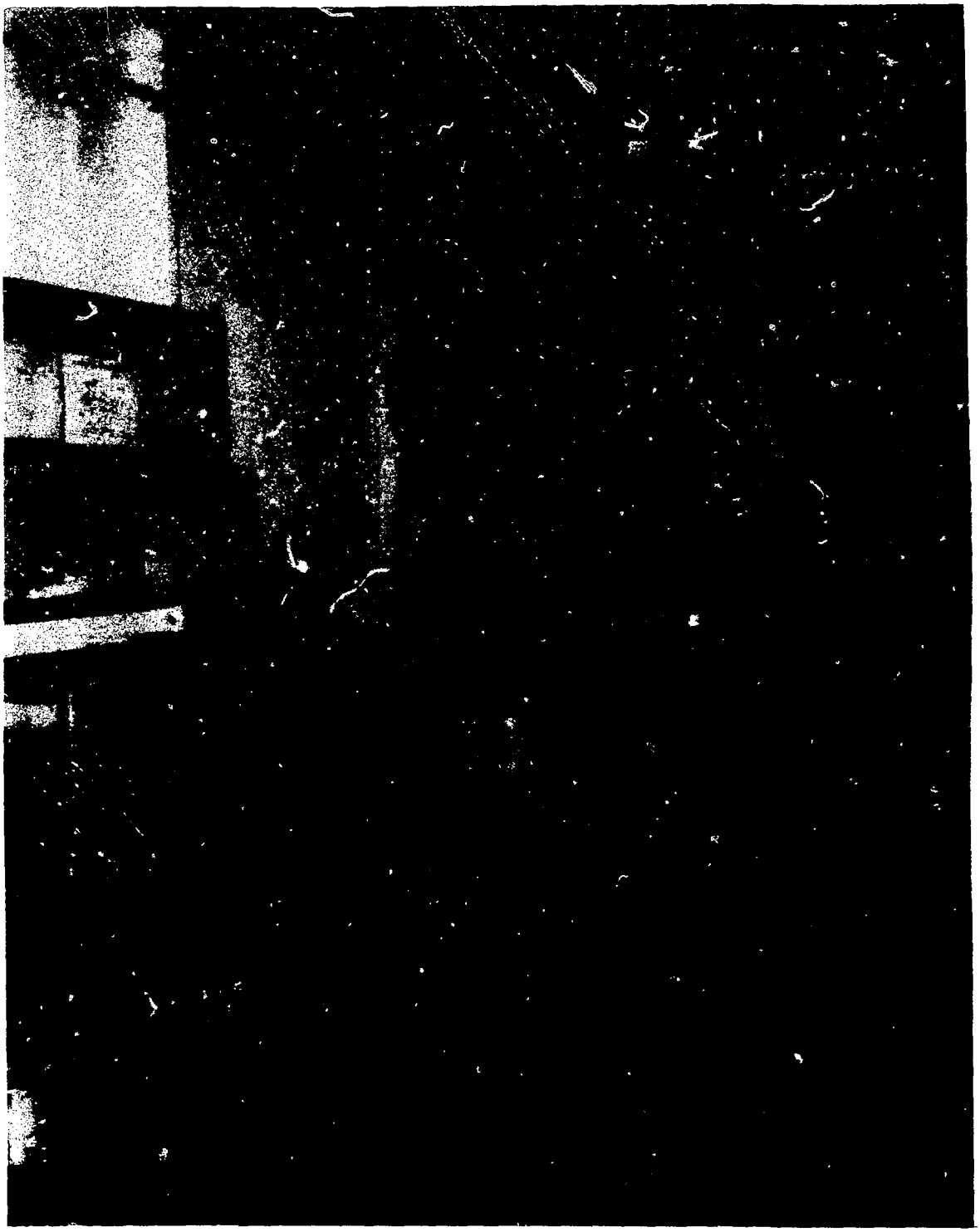


Fig. 5. Californium Shuffler assay unit installed in the scrap storage vault of the SRP reactor fuel tube fabrication facility (EG&G Neg. No. 1969).

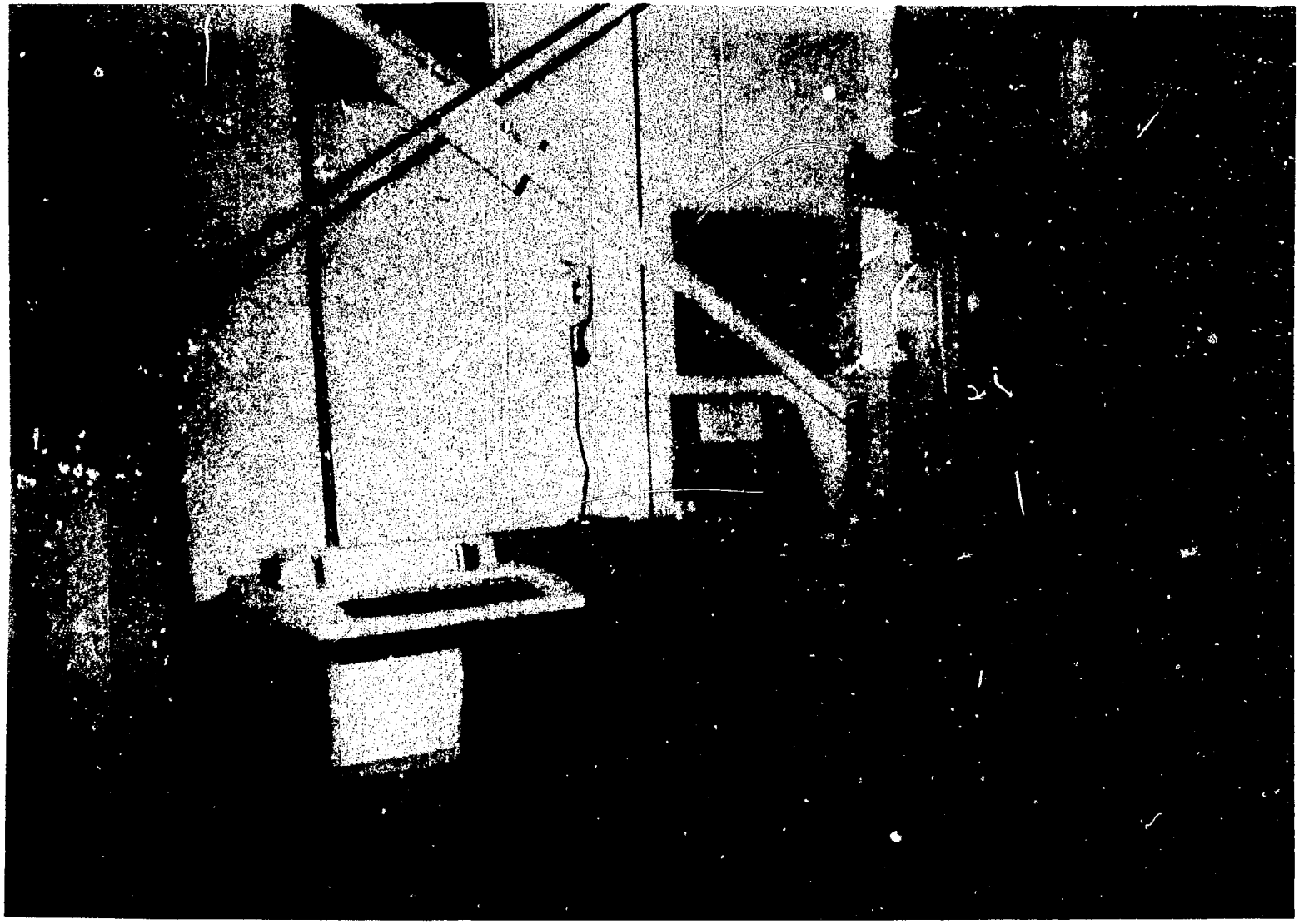


Fig. 6. Californium Shuffler electronics rack and terminals at the SRP reactor fuel tube fabrication facility (EG&G Neg. No. 1970).

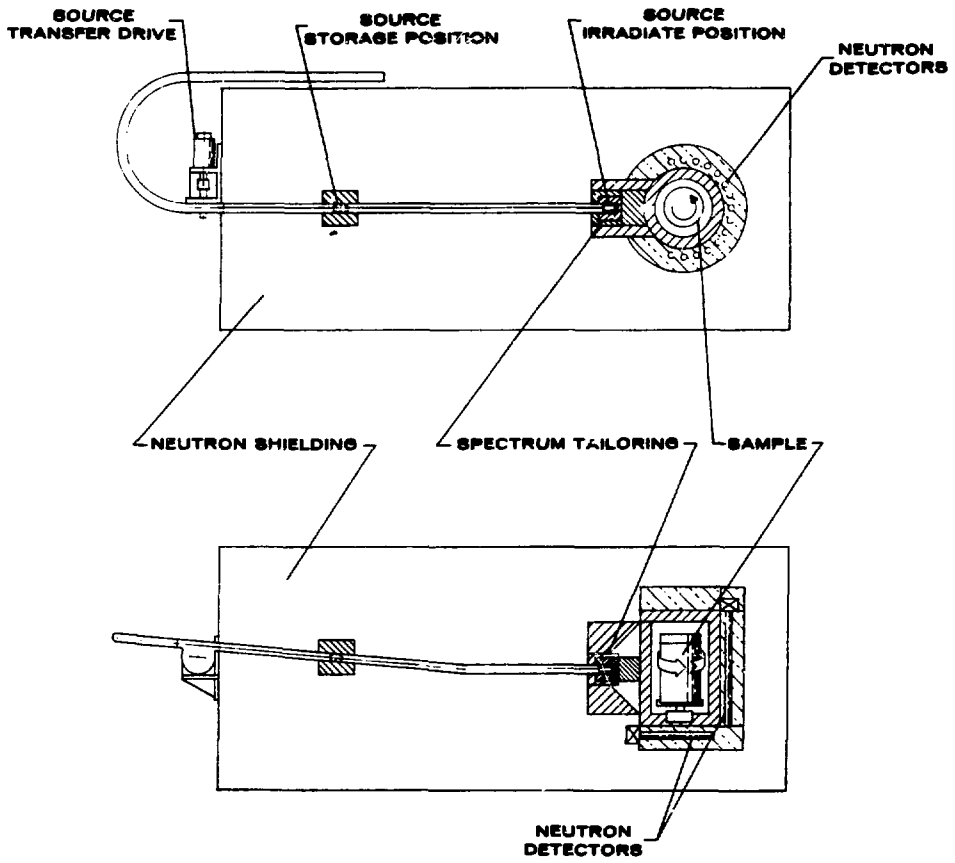


Fig. 7. Schematic diagram of the ^{252}Cf Shuffler assay unit (EG&G Neg. No. 10099).

This direct measurement of the source strength eliminates the need for including the source decay rate in the analysis.

Shielding in the storage unit is graded to minimize both neutron and gamma-ray radiation exposure. Shielding from the source position outward is first tungsten for source gamma-ray absorption, then lithium-loaded polyethylene for neutron absorption without gamma-ray emission, then boron-loaded polyethylene, and then pure polyethylene for neutron absorption and structural strength. The outer surface is cadmium for thermal neutron absorption, lead for gamma-ray absorption, and steel plate for structural rigidity. Shielding in the assay unit is similarly arranged; however, voids for motor and detector access increase the radiation leakage. Surface radiation levels are listed in Table II.

TABLE II

NEUTRON AND GAMMA-RAY EMISSION LEVELS
FROM THE ^{252}Cf SHUFFLER

Location	Neutron Dose (mRem/h)	Gamma-Ray Dose (mR/h)
Storage end		
Contact	0.5	4.0
914-mm away	0.1	0.7
Irradiation end		
Contact	2.3 (max)	19 (max)
914-mm away	0.35	2.5

A schematic diagram of the source transfer hardware is shown in Fig. 8. The motor, cable take-up tube and sensors, and the motor power supply are mounted on the Shuffler assay unit.[9] The californium source is attached to the Teleflex cable through a coupling unit. The gear driven by the stepping motor is cut with a pattern to match the helical winding of the Teleflex cable. The movement principle is similar to a rack and pinon. The gear rotates on sealed bearings mounted in a solid stainless steel block for long-term, reliable operation. The stepping motor is bidirectional with 200 steps-per-revolution or about 1 mm of cable travel per step. The controller may be a computer operated in either a high-speed accelerate/decelerate mode with the total travel distance set by the thumb wheels on the front panel, or it can be operated in a single-step mode for fine positioning of the source.

The location of the source is determined by optical sensors positioned on the cable take-up tube shown in Fig. 8. The take-up tube guides the cable between the light-emitting diode (LED) and phototransistor receiver of the optical sensor. If both sensors are blocked, the source is in the storage position because the cable is all the way out. If both sensors are unblocked, the source is in the irradiation position. A third case is when the storage sensor in unblocked and the irradiate sensor is blocked; in this case the source is in the transfer mode. The three source positions of storage, transfer, and irradiate are indicated by LED lamps on the front panel of the computer chassis. During an assay, the free end of the cable is moved to the first stepping-motor step past blocking the storage sensor for the storage position; and for the

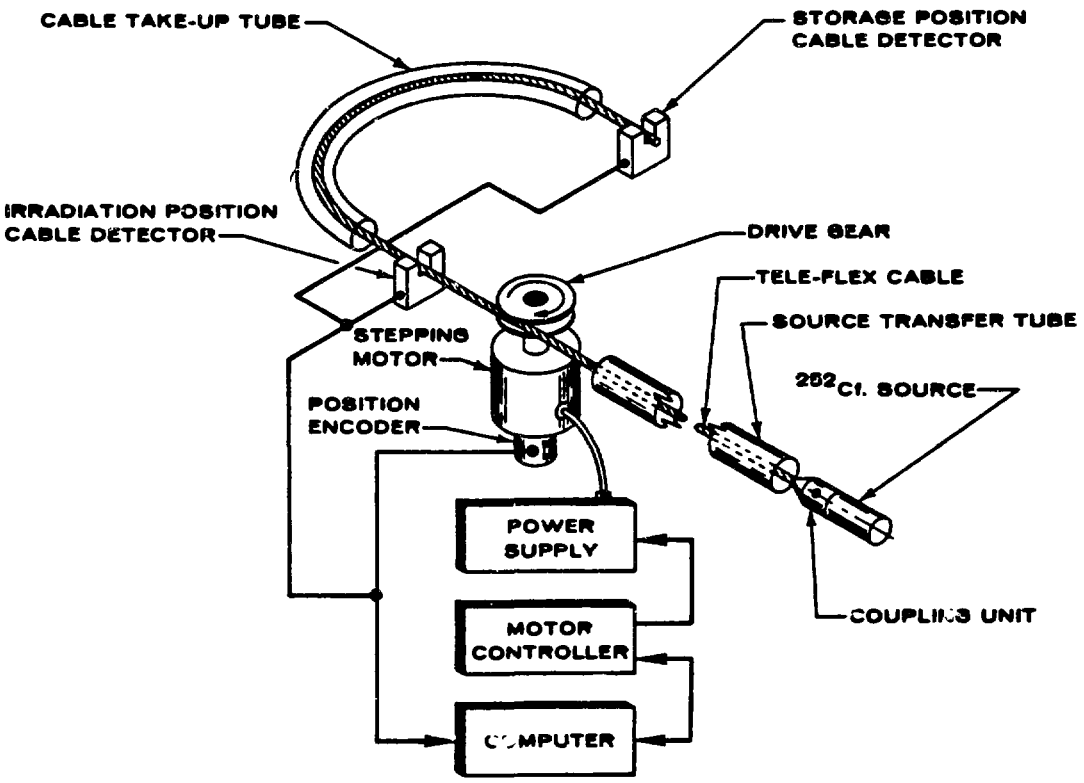


Fig. 8. Californium source transfer hardware (EG&G Neg. No. 10320).

irradiation position, the cable is moved to the point where the irradiation position sensor is just unblocked.

B. Hardware Outside the Scrap Vault

The terminals and electronics rack are shown in Fig. 6. Access to the electronics is accomplished through gasketed doors on the front and rear of the rack. Status lamps on the control panel can be viewed through a Plexiglas window on the front door of the rack. The air conditioner mounted on the side of the rack controls the temperature and humidity by shunting the air flow rather than turning the compressor motor on and off to eliminate line voltage drops and surges.[9] During normal operation, the doors on the rack are closed to prevent dust accumulation and moisture condensation.

Electronics housed in the rack include a LSI-11 microcomputer, a dual-drive floppy-disk unit, load-cell transducer, temperature transducer, stepping-motor controller, a NIM bin with high-voltage bias supplies and single-channel

analyzers for the neutron detectors, and a power conditioner for the ac line voltage.[9] A dew-point hygrometer is also mounted in the rack; however, frequent failures have resulted in "retirement in place" of this unit.

The two communications terminals used with the ^{252}Cf Shuffler are shown in Fig. 4. One terminal has a keyboard and printer, and the other has a detachable keyboard and a large-format CRT screen. The operator can interact with the microcomputer through either keyboard, and all output appears at both terminals. The printer unit provides a permanent record of the interactions between the operator and the microcomputer. The large-format CRT is for viewing by operators in the scrap vault.

C. Software

Software for the ^{252}Cf Shuffler is written in modular form using FORTRAN and MACRO programming with the LSI-11, RT-11 floppy diskette-based operating system. Machine language MACRO programming is used for direct accessing of interface modules in the LSI-11 chassis. However, the bulk of the programming uses standard FORTRAN. The software manual contains a detailed list of the steps required to operate the ^{252}Cf Shuffler, further explanations of the warning messages, a list of the routines used in the programs, and an outline for modifying the software package.[10]

Four main programs have been provided for operating the ^{252}Cf Shuffler. ASSAY is used for making assays, TEST is used for checking the operation of the Shuffler, CALIB is used for calibration, and REVIEW is used for compiling results of many assays. Normally, the operator needs to use only the ASSAY program. This program guides the operator through checking the performance of the Shuffler and making assays. Provisions have been incorporated for special assays. Malfunctions or operator mistakes can initiate error messages. In the case of a malfunction, the TEST program allows exercising individual systems to further pinpoint the origin of the failure. All data from each assay are written on a diskette to provide archival records.

III. MAINTENANCE REQUIREMENTS

The ^{252}Cf Shuffler has been evaluated in two locations by two departments at SRP. Messrs. R. V. Studley and P. N. Sand, Equipment Engineering Department (EED), conducted the preliminary evaluation in Building 723-A and Dr. R. S. Thomason, Reactor and Reactor Materials Technology Department (RRMTD),

was responsible for the evaluation in the fuel fabrication facility, Building 321-M.

A. Building 723-A Maintenance

Table III summarizes system failures that occurred during preliminary evaluation.[11] Serious failures were the hoist cable breakage and disconnection of the californium source. Breakage of the hoist cable occurred when the winch was reeling in the attachment hook and the limit switch failed to stop the winch motor at the end of travel point. The limit switch was found to be miswired into a current above the design rating, eventually causing the contacts to weld closed. The mechanical and electrical design of the limit switch was considerably improved, and the winch motor torque was reduced to below the cable breakage point. To date, no further incidents have occurred with the hoist system. In the other serious failure, the californium source unscrewed from the coupling unit (Fig. 8) leaving the source in the irradiation position because it could no longer be retracted by the stepping motor. The plug to the assay chamber was not removed and no extra radiation exposure resulted. Reattachment to the cable was done at the Savannah River Laboratory (SRL) californium facility using an improved coupling unit and attachment procedure. In order to warn an operator if such an occurrence happens again, an external gamma-ray monitor sounds an alarm if the plug is raised halfway when the source is still in the irradiate position. Also, the computer verifies a low radiation level in the assay chamber prior to requesting the operator to remove the item from the assay chamber. If high levels are detected, a warning message advising the operator to call a Health Protection Department representative is issued.[12]

The 3-g bias that occurred under certain circumstances when an empty chamber was being assayed was first observed at Los Alamos just prior to shipment of the Shuffler. To avoid postponing shipping and travel arrangements, it was decided to repair the unit at SRP. The cause of the problem was traced to a defective ^{3}He tube, and replacement solved the problem. A routine for checking for this type of problem was added to the TEST program.

The remaining problems (except for the temperature scanner) resulted from failures of commercially acquired units operated in their designed mode. These failures are indicative of the routine maintenance to be expected. In general, items believed to be highly reliable were selected for use with the Shuffler; however, occasionally items were included with unknown incidence-of-repair

TABLE III

MAINTENANCE AND MODIFICATIONS MADE TO THE ^{252}Cf SHUFFLER WHILE UNDERGOING PRELIMINARY TESTING IN BUILDING 723-A AT SRP

Problem	Corrective Action
1. Cf source disconnect	Reattachment using an improved technique and new coupling unit. Add software check for disconnect and external independent radiation alarm
2. Hoist cable breakage	Rewire and redesign limit switch. Limit hoist motor torque
3. Three gram offset on empty can assay	Replace defective ^{3}He filled neutron detector
4. Temperature scanner	Correct wiring error
5. Table rotation indicator	Replace light-coupled pair
6. Stepping motor power supply	Replace output power transistor
7. Neutron detector bias voltage supply	Repair
8. Slight source movement indicating out of storage	Improve software
9. Instability of tare weight measured by load cells	Replace load cells with higher capacity units
10. Dew point hygrometer (several failures)	New sensor-head, new cables, and new readout unit supplied by vendor (instrument subsequently abandoned in place)

records. As results of the test and evaluation program become available, certain manufacturers can be excluded from bidding, or the need for certain failure-prone components can be eliminated.

B. Building 321-Maintenance

Table IV lists the failures that occurred at Building 321-M. Most failures were minor, requiring only module exchanges or fuse replacement.[13] The stepping-motor power supply failures (Tables III and IV) are believed to have been caused by an intermittent problem in the controller. A source transfer

TABLE IV

CALIFORNIUM SHUFFLER MAINTENANCE IN BUILDING 321-M AT SRP

Problem	Corrective Action
1. Instability of tare weight measured by load cells (twice)	Replace load cells. First failure occured after 15 months operation and the second failure occurred 1 week later. Cause undetermined
2. Stepping motor power supply (twice)	Replace driver cards. Failure is believed to be caused by an intermittent problem in the controller unit. The controller was also replaced
3. Printer unit	Repaired by factory field maintenance personnel
4. Solid-state relay on hoist (twice)	Replaced
5. Computer on/off switch	Replaced
6. Source position encoder board	Replaced, additional spare provided by LASL Los Alamos
7. Error for certain load cell values	Fix software "bug"
8. Computer resets	Check for power "glitches" on ac line.
9. Floppy disk fuse	Replaced
10. Printer fuse	Replaced
11. Hoist fuse	Replaced

system being tested at Los Alamos that uses the computer for the step commands, thus eliminating the controller, has completed over a million transfers without a failure.[14] The load cells have failed more frequently than expected. Although one set of load cells lasted 15 months, a replacement set failed within a week of installation. Thus far, consultation with the manufacturer and others has not led to a satisfactory explanation of the problem.

Diagnostic messages issued by the Shuffler and familiarity of SRP personnel with the system have minimized the downtime at the fuel fabrication facility.

Most of the modules require only minutes to exchange with spare units. On the other hand, access to the interior of the Shuffler is needed for load cell replacement, and this task requires several hours.

IV. SAVANNAH RIVER PLANT MATERIALS AND CALIBRATION STANDARDS

Calibration standards are used to relate the delayed-neutron count rate to the quantity of ^{235}U in the item being assayed. For the Shuffler to realize its greatest utility, it should be able to assay as many material types as possible with the fewest standards at the highest accuracy. The Monte Carlo transport codes used to optimize the design of the assay chamber also indicated which properties of the materials affect the assay result.[1] Thus, rather than produce standards for each material category, it is only necessary to produce standards that emulate effects that influence the assay, and either interpolation or extrapolation may be used in the measurement of particular categories.

The three neutronic effects that have the greatest influence on the assay are self-shielding, multiplication, and moderation by the matrix material (aluminum in the U-Al alloy). Self-shielding occurs if the outer ^{235}U layers of the material absorb an appreciable fraction of the interrogating neutrons, resulting in the interior of the item receiving a lower neutron flux. Multiplication tends to offset self-shielding because neutrons absorbed in the outer layers produce fissions, and those neutrons can also interrogate the sample. Moderation by the item being assayed increases the response and self-shielding because the ^{235}U fission cross section increases at lower energies. The remainder of this section will describe calibration materials produced by [illegible]. The calibration procedure will be described in Sec. V.

A. Uranium-Aluminum Alloy Calibration Disks

Initially, the ^{252}Cf Shuffler was calibrated using a set of 12 U-Al alloy disks. The disks were manufactured at SRP to achieve a uniform ^{235}U content to facilitate interpretation of assay results and to minimize sampling errors for chemical analysis.[15] Each U-Al alloy disk was welded into a thin-walled aluminum can to permit handling.[16] The finished disk size is 28-mm thick by 171-mm diam with a total mass of 2 kg, of which approximately 300 g is ^{235}U.

The disks were manufactured with a uranium isotopic mixture that closely matches the isotopic mixture currently being blended for fuel tubes at SRP. Four nested cylinders were extruded to form a solid U-Al cylinder (log) from

which all disks and samples were taken. Alternate calibration disks (25-mm thick) and sample disks (6-mm thick) were cut from the log (Fig. 9). Each of the 14 sample disks were quartered. The quadrants were divided into four sets with one quadrant from each sample disk.[15] One set of quadrants was destructively analyzed at SRP and another set was analyzed at Los Alamos to obtain the uranium isotopic and uranium-to-aluminum ratio for each quadrant.[17,18] In order to arrive at a final "least squares analysis" for estimating the ^{235}U content of the calibration disks, chemical analyses of the quadrants were combined with NDA measurements of the calibration disks, made with a Van de Graaff, gamma-ray scanner, and the Shuffler.[19] (Measurements made by the Shuffler at that time could only be considered as relative because the calibration had not been established.) Overall error estimation was about 0.5% for the disks to maintain consistency among the various assay techniques. Because of the care that went into preparing the disks for eventual chemical analysis, 0.5% accuracy is the practical lower bound using state-of-the-art techniques for ^{235}U determination of SRP materials.[20]

B. High-Purity DR Ingots

As indicated in Fig. 1, DR ingots are the final product of the scrap recovery operation before the material is returned to the process stream. Thus, in terms of fuel tube product quality control, the assay of DR ingots is the most useful of the scrap measurements. A typical DR ingot is about 50- to 100-mm high and 160-mm in diameter with a total mass of 1-6 kg. Depending upon the aluminum-to-uranium ratio and size, the ingots may contain between 0.1 and 1 kg ^{235}U (Ref. 3). In order to test the disk calibration, a set of carefully prepared DR standards was produced. Well characterized uranium and pure aluminum were melted to produce ingots. The disk standard calibration yielded results consistent with charge makeup and chemical analysis of samples drawn during casting.

The uranium isotopic sensitivity was investigated with ingots made from fully enriched (93% ^{235}U) and depleted (0.2% ^{235}U) uranium as well as with enrichments close to that currently being blended at SRP. Ingots were also produced with as wide a range as practical in the aluminum-to-uranium ratio. The highest uranium content was 35% by weight, and the lowest content was 7% uranium by weight.

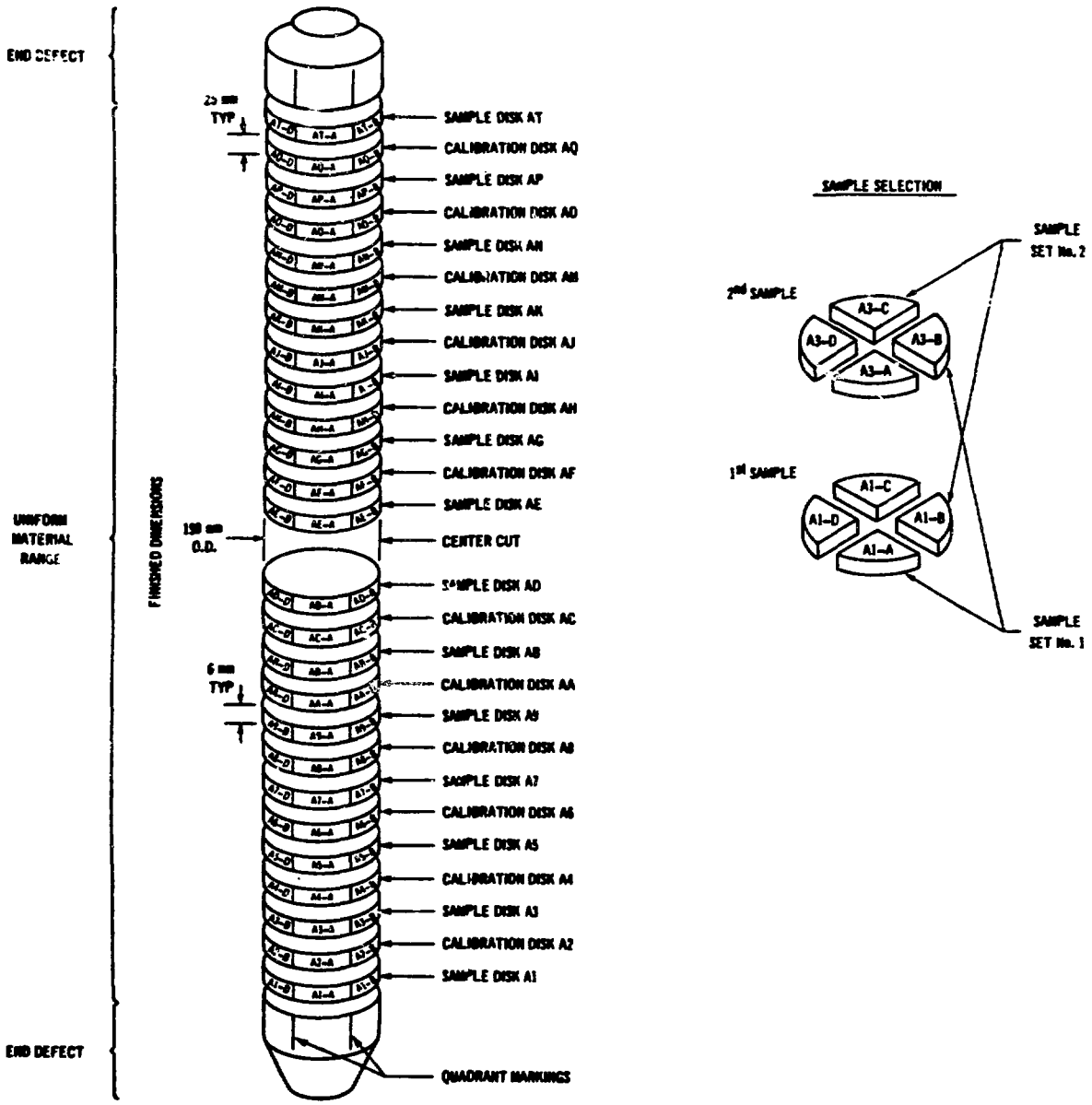


Fig. 9. Relative locations of sample and calibration disks cut from the extruded U-Al alloy log (EG&G Neg. No. 10321).

C. Chip Standards

Uranium-aluminum billets cast for production melts are machined and cut to size prior to extrusion. Machining and cutting operations produce chips that are recycled as scrap.[2] Saw chips are granular and have about half the alloy

density. Lathe chips are more variable in character depending upon the per cent uranium in the billet. High uranium content items produce a brittle alloy, and the chips are small (3 mm × 10 mm) and have about 20% of the alloy density. On the other hand, low uranium content billets result in long spiral turnings. Depending upon the vigor with which these chips are packed into the scrap cans, their density can range from 10 to 20% of the alloy density.

In order to obtain well-characterized chips, homogenous hollow U-Al alloy cylinders (cores) were cast from the same melts as the DR ingots, described in the previous section. The cores were machined to produce lathe chip standards. These standard chips were assayed by the Shuffler in both a fluffed and compacted state. A number of standards could be produced from each full can of chips simply by filling the can to be assayed to various levels. The ^{235}U content could be determined from the known chip weight, the uranium isotopic composition, and the uranium weight fraction of the core from which the chips were cut.

D. Flux Material

Flux materials contain some of the lowest purity and lowest ^{235}U-content items assayed by the Shuffler. The purpose of the flux is to reduce uranium oxides so that the uranium will dissolve in pure aluminum added to the flux. Thus, attempting to produce a "leached flux" standard is not practical because the uranium for the most part will separate from the flux.

An alternative approach was used to obtain flux material with reasonably well estimated ^{235}U content. The estimation process consisted of assaying a can of flux material in the Shuffler and recording the delayed-neutron response. Small pieces of U-Al alloy (chips) were added to the can and the assumption made that the response per gram ^{235}U from the additional chips would be about the same as that from the ^{235}U initially in the flux material. This theory was tested by placing chips at different positions within the can. In all cases the delayed-neutron responses were in satisfactory agreement, indicating that the chip response was probably quite similar to that of the ^{235}U in the flux. Data were obtained with successive additions of chips. By assuming the same response per gram ^{235}U of the chips, the quantity of ^{235}U in the flux could be calculated from the delayed-neutron response of the flux without any additional chips.[21] A later, general calibration based upon corrections for the ^{235}U density and quantity of matrix material agreed very well with the above calibration result.

V. CALIBRATION PROCEDURE

A. Theory

The disks produced by SRP are the primary standards for calibration of the Shuffler. These disks are similar in composition to most U-Al alloys in the fuel fabrication process, and stacking the disks simulates scrap of different heights. The specially prepared DR ingots and chips produced from cores from the same melts are used to correct for differences between the disks and the various scrap material categories.[1]

Data collected during an assay include the background and delayed-neutron counts; the interrogation-flux intensity; the source transfer, irradiation, and count times; and the weight of the item. The delayed-neutron count rate is corrected for background counts. The height of the item is estimated from the ratio of delayed-neutron counts in the side detector banks to the total delayed-neutron counts (bottom plus side banks). The height estimation is the key to determining correction factors because the sample geometry is then known. (The diameter of the scrap cans and most ingots is roughly the same.) The delayed-neutron flux is normalized to the observed interrogation flux to account for the californium source decay. Thus, no source half-life correction is required. The total weight of the item provides information needed to estimate moderation corrections based upon the amount of matrix material.

The calibration of the Shuffler response to the stacked disks is nearly linear with small quadratic and cubic terms in the mass to account for changes in coupling to the interrogation flux monitor detectors associated with the stack height of the disks.[22] The mathematical expressions for the correction factors are arranged so as to give a zero correction value when the item resembles the stacked disks and a linear correction as a particular parameter deviates from that of the disks.

B. Special Cases

The Shuffler was optimized for response uniformity from either ingots or loose materials. Two assumptions that went into the optimization calculations were that material would fill the cans from the bottom up and that it would be somewhat homogeneous. Somewhat homogeneous means that the can does not consist of a single lump of ^{235}U material surrounded by matrix material or the scrap can does not contain two different material categories segregated within the can. An example of the latter case would be a DR ingot placed on top of lathe chips.

Intermixing of scrap material types is not permitted by SRP operating procedures. Floor sweepings could have the ^{235}U concentrated in a localized region. However, sweepings generally have uranium alloy lumps dispersed throughout the volume.

In any case, checks have been placed in the software to ensure that the material appears to be reasonably uniform. In cases where the material appears to be nonuniform, the operator is advised to repackage the waste into two cans. Because the Shuffler has a reasonably uniform response from items in the bottom half of the can, regardless of position, packaging the waste in two cans will improve the assay accuracy.

In actual operation, few scrap cans were found to be so nonuniform that the computer prompted the operator to repackage material. To test this warning feature, the U-Al calibration disks were stacked with pure aluminum disks to obtain extreme cases. If a single calibration disk is placed at the bottom of a scrap can and the remainder filled with aluminum, or if four or more pure aluminum disks (100-mm thickness) are placed at the bottom of the can and then any combination of calibration disks with aluminum, a warning is issued.

The calibration includes data from assays of a single calibration disk interspersed with pure aluminum. Assays of similarly distributed items can be analyzed with this special calibration with the REVIEW program. However, the analysis is not available to the operator on a routine basis. In cases where uniformity or sample height criteria are not met, the operator is not given any ^{235}U mass value.

C. Procedure

Calibration of NDA instruments is essential for accurate assays. Some measurement techniques require frequent and even daily calibration to maintain reliable results. On the other hand, the Shuffler uses an inherently stable technique, and months of operation using the same calibration are possible. Long-term stability of the instrument allows calibration standards to be assayed over an extended period of time. In addition, as standards become available, the entire set of standards does not have to be remeasured because all previous measurement results are available on disk files.

Calibration of the Shuffler is accomplsihed with a program called CALIB. The input to this program is a list of calibration data file names and the ^{235}U content of each item. The output of the calibration program is a file containing the input and the best fit value for each calibration constant and its

uncertainty. The calibration constants are available on the disk for data-analysis subroutines.

Three types of assays are used in the calibration procedure. First, there are the calibration disk results. Ten assays are required: first a single disk, then two disks, with a disk added for each subsequent assay until a total of ten disks are assayed at once. These data provide the basic calibration and the constants used by the correction factors to relate results from a wide range of materials back to the standard disks. Comparisons with other materials is made through the ^{235}U content per unit height, the total mass per unit height, or the total mass for a sample of a given height.

The next 10 assays provide data for determining possible effects of various nonuniform mixtures of U-Al alloy and pure aluminum. These data are assays of a single U-Al alloy disk and nine aluminum disks. For the first data point, the U-Al alloy disk is under all nine aluminum disks, then over one and under eight, then over two and under seven, until for the tenth data point, the U-Al disk is on top of all nine aluminum disks. Data obtained with the nonuniform distribution is available for interpreting results or understanding the behavior of the Shuffler in unusual assay situations.

The last data group used in the Shuffler calibration may contain up to 20 items, and the data are used to determine the correction factor coefficients. There are no restrictions on the data that can be used for this purpose; however, items with the greatest variation in ^{235}U density and uranium-to-aluminum ratio will do a better job of determining correction factor coefficients. The items include the specially prepared DR ingots and saw and lathe chip standards. In the lathe chip case, assay data from both a fluffed and a compacted state are included.

A list of the calibration standards and "best fit" results is given in Table V. The nonuniform mixture of uranium and aluminum is included for completeness; although, this material is not used for determining the calibration for other materials. The correction factor is presented in a multiplicative form as it is used in the formulas. Thus, a unity value (1.000) is equivalent to no correction. Data for the stacked standard disks (the first 10 items in Table V) show small corrections, as they should. The last 20 times in the table show correction factors up to 6.4% (1.064) indicating that the magnitude of the response variation among the various SRP scrap material categories is not excessively large. Ignoring the intentionally nonuniform material (items 11-20 in

TABLE V

CALIBRATION STANDARDS AND THEIR BEST FIT RESULTS

Item	Assigned ^{235}U (g)	Assayed ^{235}U (g)	Difference (g)	Chi-Square Contribution	Correction Factor	Comments
1	282.2(0.9)	278.2(6.2)	-4.0(6.2)	0.40	1.015	1 U-Al disk
2	563.5(1.8)	557.3(7.0)	-6.2(7.2)	0.74	1.002	2 U-Al disks
3	844.7(2.7)	852.5(7.5)	7.8(8.0)	0.94	0.994	3 U-Al disks
4	1 126.7(3.7)	1 131.8(7.1)	5.1(8.0)	0.40	0.998	4 U-Al disks
5	1 409.8(4.6)	1 411.6(6.8)	1.8(8.2)	0.05	1.001	5 U-Al disks
6	1 694.8(5.5)	1 698.0(6.8)	3.2(8.7)	0.13	1.001	6 U-Al disks
7	1 977.6(6.4)	1 976.3(7.6)	-1.3(9.9)	0.02	0.999	7 U-Al disks
8	2 259.8(7.3)	2 253.7(8.1)	-6.1(10.9)	0.31	1.000	8 U-Al disks
9	2 541.4(8.2)	2 528.1(10.2)	-13.3(13.1)	1.03	1.000	9 U-Al disks
10	2 818.6(9.1)	2 832.2(18.0)	13.6(20.2)	0.45	1.000	10 U-Al disks
11	281.3(0.9)	283.1(2.1)	1.8(2.3)	0.62	a)	U-Al/9-Al disks
12	281.3(0.9)	314.8(18.1)	33.5(18.1)	3.43	1.049	Al/U-Al/8-Al disks
13	281.3(0.9)	291.5(6.4)	10.2(6.5)	2.48	1.088	2-Al/U-Al/7-Al disks
14	281.3(0.9)	299.2(4.6)	17.9(4.7)	14.30	1.079	3-Al/U-Al/6-Al disks
15	281.3(0.9)	280.6(3.9)	-0.7(4.0)	0.03	a)	4-Al/U-Al/5-Al disks
16	281.3(0.9)	286.0(4.1)	4.7(4.2)	1.28	a)	5-Al/U-Al/4-Al disks
17	281.3(0.9)	287.9(5.0)	6.6(5.1)	1.69	a)	6-Al/U-Al/3-Al disks
18	281.3(0.9)	289.8(5.4)	8.5(5.4)	2.45	a)	7-Al/U-Al/2-Al disks
19	281.3(0.9)	276.1(4.4)	-5.2(4.5)	1.32	a)	8-Al/U-Al/1-Al disks
20	281.3(0.9)	265.3(8.1)	-16.0(8.1)	3.88	a)	9-Al/U-Al disks
21	1 650.3(5.0)	1 671.6(11.8)	21.3(12.8)	2.77	0.997	Saw chips
22	186.6(3.0)	192.0(2.3)	5.4(3.8)	2.04	1.061	7% U-Al ingot
23	467.1(5.0)	460.2(4.3)	-6.9(6 6)	1.10	1.038	16% U-Al ingot
24	775.3(7.0)	768.5(6.8)	-6.8(9.8)	0.49	1.003	24% U-Al ingot
25	966.2(9.0)	973.7(11.9)	7.5(14.9)	0.25	0.976	30% U-Al ingot
26	1 224.3(11.0)	1 220.0(15.6)	-4.3(19.1)	0.05	0.967	35% U-Al ingot
27	239.6(5.0)	237.1(2.6)	-2.5(5.6)	0.19	1.053	Compacted chips
28	239.6(5.0)	242.3(2.4)	2.7(5.5)	0.24	1.045	Fluffed chips
29	354.2(5.5)	344.1(3.0)	-10.1(6.2)	2.61	1.042	Compacted chips
30	354.2(5.5)	352.5(2.8)	-1.7(6.2)	0.08	1.032	Fluffed chips
31	560.9(6.0)	559.0(3.8)	-1.9(7.1)	0.07	1.022	Compacted chips
32	560.9(6.0)	552.5(4.4)	-8.4(7.4)	1.26	1.014	Fluffed chips
33	643.0(7.0)	631.4(4.3)	-11.6(8.2)	2.01	1.018	Compacted chips
34	643.0(7.0)	639.2(5.0)	-3.8(8.6)	0.19	1.012	Fluffed chips
35	127.3(3.0)	131.8(2.3)	4.5(3.8)	1.45	1.064	Chips, 25-mm high
36	208.5(4.0)	211.7(2.6)	3.2(4.8)	0.43	1.056	Chips, 51-mm high
37	345.4(7.0)	350.4(3.5)	5.0(7.8)	0.40	1.044	Chips, 76-mm high
38	345.4(7.0)	349.9(3.0)	4.5(7.6)	0.35	1.039	Chips, 114-mm high
39	397.2(8.0)	404.0(3.4)	6.8(8.7)	0.61	1.039	Chips, 102-mm high
40	397.2(8.0)	404.0(3.1)	6.8(8.6)	0.62	1.034	Chips, 127-mm high

[a]Determined by the Shuffler to be nonuniform.

Table V), the Chi-Square value is 21.7 for the remaining 30 items. Thus, the calibration is reasonable, and a single calibration is adequate for at least the ingots and chips generated during the reactor fuel tube fabrication.[23-27] Use of a single calibration is a significant benefit because the need to produce several standards for each material category is eliminated. In addition, the possiblity of an incorrect analysis because the wrong calibration curve was selected is avoided.

VI. ASSAY PERFORMANCE

A. Stability

Consideration of the stability of the ^{252}Cf Shuffler will be separated into three categories: short-term stability, long-term instrument stability, and long-term consistency of SRP materials. Short-term stability refers to how well the estimated statistical uncertainty in the assay agrees with the observed variation in the assay results. Results were measured during a short time interval so that slow instrument drifts are negligible. Long-term instrument stability refers to systematic drifts in the assay due to slow changes such as component aging or the loss of neutron source intensity due to radioactive decay. Long-term consistency refers to change in the materials fabricated at SRP due to the slowly changing composition of recycled uranium blended into the fuel tube alloy.

Statistical uncertainty for each assay is related to the total number of delayed-neutron counts. Two estimates of the statistical precision are made for each assay, and the larger is chosen. The first uncertainty estimate is based on the random error computed for the square root of the number of observed counts. The assay uncertainty is then determined by standard error propagation techniques from the counting statistics.[27] The second error estimate uses the standard deviation of the individual assay cycle results corrected to equilibrium rates on a cycle-by-cycle basis.[28,29] The larger uncertainty is then taken as the estimated value. The cycle-by-cycle estimate gives about the same error estimate as the counting statistics unless a small malfunction or background rate change occurs during the assay. For example, if an exceptionally strong neutron-emitting item were moved in the vicinity of the Shuffler, then the background rate could be changed during the assay. Unexpected systematic variations are then detected by the cycle-to-cycle error estimate, and small shifts

in the assay are accommodated by increasing the final result uncertainty.[29] Table VI shows statistical analysis of data from 30 replicate assays. The short-term stability is consistent with the expected statistical uncertainty, because the standard deviation of the full 30 assays is reasonably consistent with the expected value estimated from individual assays.

TABLE VI

STATISTICAL ANALYSIS OF THE DELAYED-NEUTRON RESPONSE FOR 30-SEQUENTIAL REPLICATE ASSAYS

Sequence Number	Response	Error Estimation	Cycle-to-Cycle Error Estimation	Assigned Error Estimation	Response Deviation from Mean (%)
1	14 457.5	32.4	33.0	33.0	0.16
2	14 431.3	32.5	37.8	37.8	-0.02
3	14 436.9	32.4	38.4	38.4	0.01
4	14 414.4	32.8	34.9	34.9	-0.14
5	14 438.2	32.5	29.0	32.5	0.02
6	14 505.1	32.7	23.9	32.7	0.49
7	14 456.5	32.2	38.3	38.3	0.15
8	14 428.2	32.6	34.8	34.8	-0.05
9	14 475.5	32.2	32.0	32.2	0.28
10	14 395.6	32.3	30.1	32.3	-0.27
11	14 421.4	32.5	28.4	32.5	-0.09
12	14 386.9	32.5	31.3	32.5	-0.33
13	14 388.7	32.1	35.2	35.2	-0.32
14	14 431.2	32.1	27.9	32.1	-0.03
15	14 353.5	32.2	28.3	32.2	-0.56
16	14 411.5	32.2	37.0	37.0	-0.16
17	14 456.2	32.3	34.0	34.0	0.15
18	14 513.1	32.8	32.8	32.8	0.54
19	14 444.8	32.4	34.3	34.3	0.07
20	14 468.7	32.6	31.2	32.6	0.23
21	14 463.0	32.4	23.7	32.4	0.19
22	14 453.3	32.5	33.2	33.2	0.13
23	14 408.4	32.6	33.4	33.4	-0.18
24	14 393.7	32.6	32.9	32.9	-0.29
25	14 494.0	33.0	32.8	33.0	0.41
26	14 440.1	32.5	31.8	32.5	0.04
27	14 460.7	32.5	35.2	35.2	0.18
28	14 462.1	32.8	33.7	33.7	0.19
29	14 381.2	32.8	29.9	32.8	-0.37
30	14 374.1	32.5	30.4	32.5	-0.42
Mean Value	14 434.9	32.5	32.3	33.8	0.00
Std. Dev.	39.1	0.2	3.7	1.9	0.27

Long-term stability has been observed for over a year in the reactor fuel fabrication facility. The SRP procedure has been to make an assay of six standard disks at the beginning of each assay session.[30] A comparison is incorporated into the software to check the assay against a specified previous result to assure that the Shuffler is working properly.[10] The accumulated set of these assays is used to determine the magnitude of long-term drifts. Table VII shows the accumulated assay results on a monthly basis. The standard deviation of the assay results is 0.4%. The trend of the monthly data has been toward an increasing response with an average rate of about 0.1% per month. Cause of the drift has not been definitely determined, but suggested causes include a change in neutron pile-up events in the flux monitor detectors compared to the delayed-neutron detectors as ^{252}Cf source intensity decreases, slight changes in the source-to-sample coupling geometry when transfer hardware is disassembled and reassembled, changes in flux monitor position when the load cells are replaced, electronic drifts, and shifts in the source transfer time due to drifts in the controller unit. After reviewing the assay data and comparing with operation and repair records, the possible cause most consistent with the data is drifts in the transfer time. To eliminate this cause, each transfer time is now recorded and used by the analysis codes to correct the assay. A second possible cause, consistent with some of the data, is the shift in position of the small ^{3}He-filled neutron detectors used to monitor the neutron flux from the californium source during irradiation. These two neutron detectors are located in the base of the assay chamber and are held in notches cut in the Boral, cadmium, and iron side wall by a stainless steel liner. To test "worst" case configurations, the liner was removed, and wedges were inserted to move the flux monitors as far out of position as possible while still being able to reinsert the liner. A shift as large as 10% was observed. To eliminate possible movement of the flux monitors when the liner is removed to replace the load cells, a rigid mounting for these detectors was made by attaching the detector assembly to the assay chamber base. Final positioning gave a response within 0.1% of the June 1979 value.

In any case, besides remedying the cause of the drift, a solution would be to recalibrate the Shuffler before the drift becomes appreciable. Thus, recalibration about every six months would be adequate when coupled with monitoring the replicate assays. The present procedure calls for assaying the standard

TABLE VII

SUMMARY OF LONG-TERM ASSAY REPEATABILITY

Month and Year	Number of Assays	Average Response
June 1979	8	28 408.2
July	13	28 431.6
August 1979	5	28 379.9
September 1979	6	28 401.4
October 1979	13	28 464.2
November 1979	13	28 444.1
December 1979	8	28 425.4
January 1980	1	28 558.6
February 1980	2	28 696.2
March 1980	3	28 608.5
April 1980	12	28 622.4
May 1980	11	28 689.4
June 1980	11	28 709.2

Average Response 28 522.1

Standard Deviation 122.1 (0.4%)

Drift Coefficient 0.1% per month

disk set at the start of each measurement session. At the close of each session, one of the calibration standards could also be assayed. In a period of 2 to 3 months, there would be ample time to assay up to the maximum 40 standards allowed by the calibration program. Thus, the calibration could be fully updated about every three months without appreciably impacting daily operation at the fuel fabrication facility.

Long-term changes in the uranium-aluminum alloy used at SRP result from blending recycled uranium of slowly changing composition with highly enriched (93% ^{235}U) uranium. The result of this process yields a uranium isotopic mixture with a gradually decreasing ^{235}U fraction and a gradually increasing ^{236}U fraction. Figure 10 shows the ^{234}U, ^{236}U, and ^{238}U fraction as a function of the ^{235}U enrichment. The change in the isotopic composition is a controlled, step-wise process. The current uranium enrichment is 60%, and enrichments of 40% ^{235}U are anticipated about 1990 (Ref. 3).

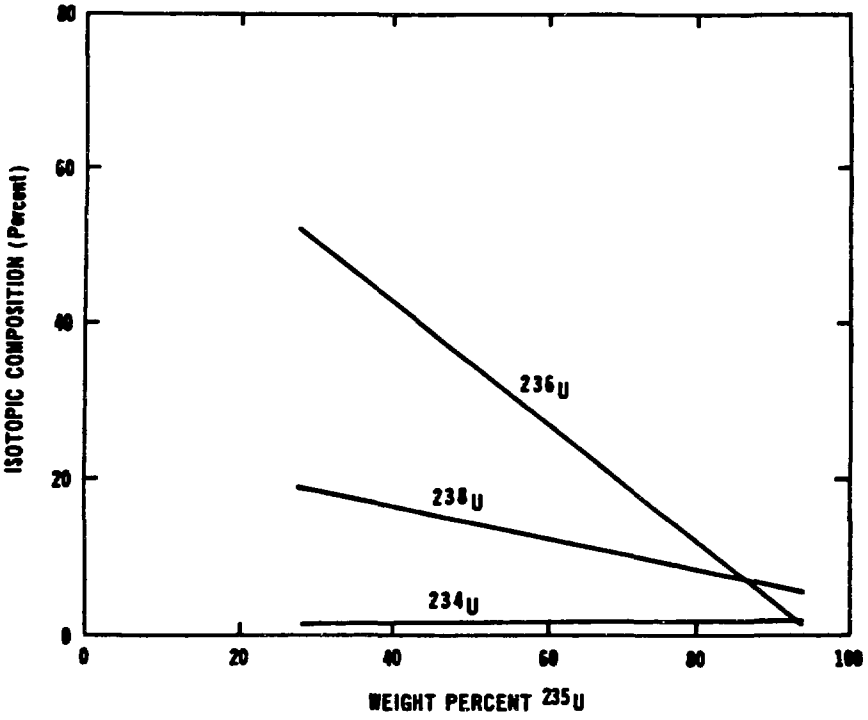


Fig. 10. Isotopic composition of uranium blended at SRP (EG&G Neg. No. 10018).

In order for the fuel tubes to have the desired reactivity, the alloy requires a higher uranium content to compensate for the lower ^{235}U fraction. Correction factors are already in use to adjust the Shuffler calibration for the uranium-to-aluminum ratio. However, the isotopic mixture also affects the response of the instrument. The Shuffler response is calibrated for the ^{235}U content of the standards. Because ^{235}U is the only fissile isotope in the SRP alloy, the response is about 98% from ^{235}U. The even isotopes (^{234}U, ^{236}U, and ^{238}U) contribute to the response when a neutron above their respective fission threshold causes a fission. These high-energy neutrons come from the ^{252}Cf source and from fissions of the ^{235}U within the item. For 10 standard disks assayed by the Shuffler, the even isotope response is about equally divided between the two causes with each contributing about 1% of the ^{235}U response.[1] A direct measurement of the isotopic mixture is in principle possible by identifying the ratio of precursor groups contributing to the delayed neutron signal. In practice, the difference is so slight as to make the technique impractical.[31]

The best approach to avoid biased results because of the isotopic mixture is to recalibrate the Shuffler with materials of the same isotopic composition as those currently being processed. For a given set of standards, the useful calibration range is valuable for determining when new calibration standards will be required and the magnitude of systematic errors for different isotopic compositions. Correction factors could be added to account for slightly different compositions.

The first response estimate from even isotopes was based on the Monte Carlo code results for the various isotope fission rates and the delayed-neutron emission rates. Unfortunately, no measurements for delayed-neutron yield from ^{234}U or ^{236}U have been made, and the yields have to be estimated from an empirical formula.[5] For a benchmark comparison, six sample bottles of UF_6 ranging from 3.001% to 97.65% ^{235}U were assayed.[32] The results yielded a ^{238}U response that was 5% of the ^{235}U response for equal quantities of each isotope. The Monte Carlo results combined with the empirical formula agreed with the observed responses. However, if the actual delayed-neutron yields and precursor time dependence data for Shuffler assay cycle is used, then the calculated ratio is 3%.

In order to test the Shuffler dependence on the uranium isotopic fraction, a set of DR standards was prepared. Uranium-235 enrichments of 48, 60, 76, and 93% were cast in DR molds. The ingots were prepared to a uniform height and uranium weight fraction. Figure 11 shows the difference between ^{235}U content measured by the Shuffler and the charge makeup of the castings. The error bars represent the combined uncertainty in the Shuffler calibration and the ^{235}U content of the standards. Data were taken for ten 30-cycle assays of each standard so that statistical fluctuations do not contribute to the error bars shown in Fig. 11. In addition to data obtained with the standards, the calculated isotopic dependence is plotted in Fig. 11. We can immediately note that the measured and calculated isotopic dependence do not agree for the higher enrichment data at 76 and 93%, while the agreement is good at the 48 and 60% enrichments. Although the Shuffler accurately determines the ^{235}U content for the higher enrichment items, the correct value is actually a result of an over compensation by the self-shielding correction factor. The set of DR standards made to check the isotopic dependence of the Shuffler were designed to eliminate geometric effects and uranium weight fraction effects; however, the changing self-shielding by various quantities of ^{235}U in each standard produced effects

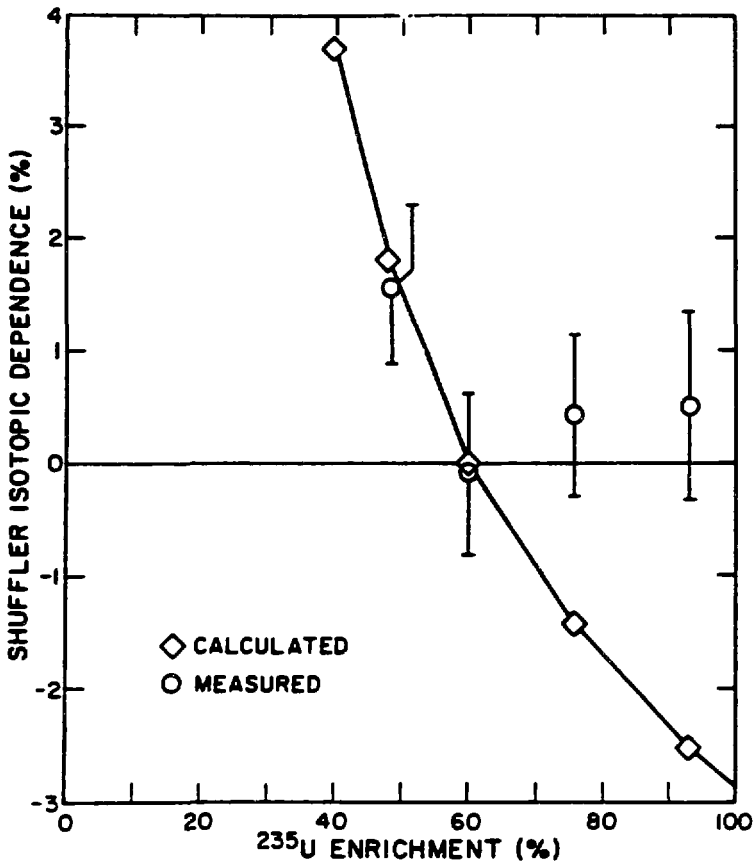


Fig. 11. Percentage error in the assayed ^{235}U content for the Shuffler calibrated with 60% enriched uranium (Los Alamos Neg. No. 80-9354).

comparable to those expected for the enrichment. An additional set of standards at about 3% uranium weight fracton cast in core molds is being considered. In the meantime, the Monte Carlo results combined with the empirical formula still are the best guess for the Shuffler enrichment dependence for the SRP uranium alloy. Calculation predicts a 1% bias in assay results when the SRP ^{235}U enrichment is decreased to 52%.[33] Thus, when the ^{235}U enrichment blended at the SRP fuel fabrication facility is reduced to about 50%, it is recommended that a new set of calibration standards be produced for the Shuffler. For enrichments between the current value of 60% and 50%, a linear correction based upon the known enrichment and the accurately determined isotopic dependence should be adequate.

B. Accuracy

1. DR Ingots. Figure 12 shows a summary plot of DR ingot measurements. For the most part, the ingots were produced in scrap recovery operation at the SRP reactor fuel fabrication facility.[34] The ^{235}U value assigned to the ingots is based upon samples drawn from molten alloy during casting. The samples are destructively analyzed to determine the ^{235}U enrichment by mass spectrometry, and the uranium weight fraction is determined by chemical analysis using the Davies-Gray method.

The uncertainty assigned to the destructive analyses for DR ingots is plus or minus one percentage point in the uranium weight fraction and 0.1% relative error in the ^{235}U isotopic. Thus, an item containing 10% uranium by weight has a 10% uncertainty in the uranium content, and an item containing 20% uranium by weight has a 5% uncertainty in the uranium content. Errors assigned to DR ingots typically range from 4 to 12% (one standard deviation).

The uncertainty assignment for the DR ingot destructive analyses results in a nearly constant ^{235}U uncertainty of about 30-g per item. The 30-g ^{235}U uncertainty is indicated in Fig. 12. Assays made by Shuffler are generally in good

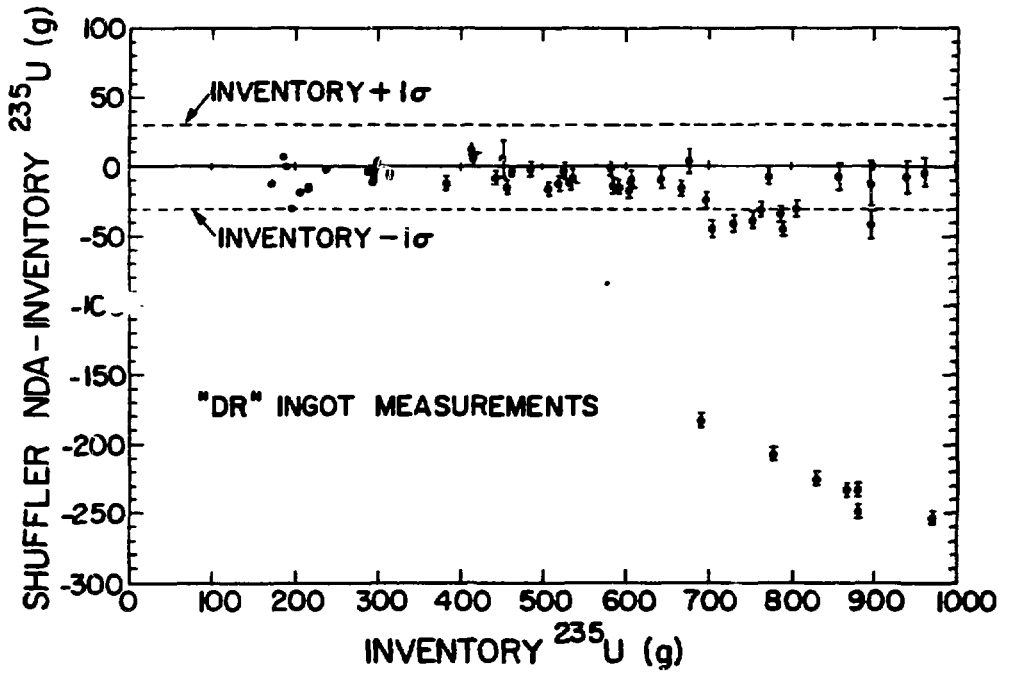


Fig. 12. Difference between the inventory ^{235}U content and the NDA measurement made by the Shuffler for DR ingots (Los Alamos Neg. No. 80-7458).

agreement (less than one standard deviation) with inventory values determined by chemistry. However, some notable exceptions are quite obvious in the lower right-hand corner of the data plotted in Fig. 12. DR ingots represented by those data were poured from two melts made on the same day. The data also extrapolate back toward a zero intercept value indicating a possible mistake in either the uranium weight per cent or the ^{235}U enrichment. By the time the discrepancy between the Shuffler value and the chemistry results had been noted, all but one DR ingot from each melt had already been recycled into a production melt. The two remaining ingots were melted and resampled. The Shuffler and chemical assays confirmed the earlier Shuffler values. Other data that were initially found to be discrepant were traced to "bookkeeping" errors or attributed to erroneous chemistry results.[30]

2. Saw Chips and Lathe Chips. A saw chip standard was produced from a single extruded cylinder (log) by repeatedly cutting it until a full can of chips was obtained.[35] Careful preparation went into fabricating the U-Al log so that the ^{235}U content could be determined directly from the weight of the chips. Overall accuracy for this standard is estimated to be 0.3%. Because of the detailed monitoring of the cutting phase of fabricating this standard material, the quantity of alloy removed during sawing operations is more precisely known. This new data has been incorporated into the SRP material accounting procedures. The saw chips are item 21 in the calibration results given in Table V. The quantity of ^{235}U in the standard is 1605.0 ± 5.0 g. The value measured by the Shuffler is within 1.3%.

Lathe chip standards were turned from cores poured from the same melts as some of the DR standards. About 48 lathe chip standards were assayed for both calibration and test measurements. The large number of items resulted from scrap cans being filled to various heights and the chips being either fluffed up or tightly compacted into the cans. Chips were cut from cores of different uranium weight percentage because the size and character of the turnings changes with the uranium fraction (Sec. IV. C.) and the delayed-neutron response of the Shuffler has a slight dependence on the uranium fraction in the alloy.

Figure 13 displays data from lathe chip measurements. The error bars indicated are from the Shuffler measurements alone, and they include statistical error and calibration uncertainties. The uncertainty in inventory values is about 0.5 to 1%. Data with the same inventory mass value result from compacting the chips and reassaying. Overall agreement for the chips was 1.3%.

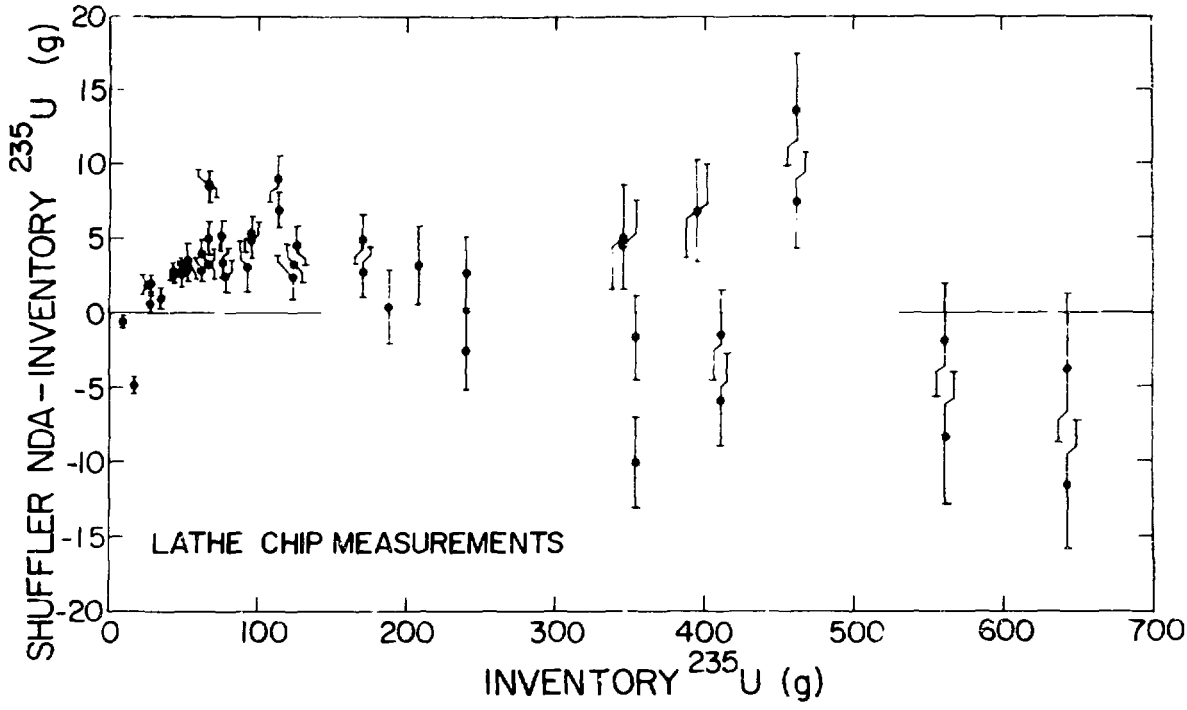


Fig. 13. Difference between the inventory ^{235}U content and the NDA measurement made by the Shuffler for lathe chips turned from core standards (Los Alamos Neg. No. 80-7459).

3. Risers. Risers are material cut from the top end of castings produced from production melts (Fig. 1). Risers are broken with a hydraulic press and stored in scrap cans prior to being added to a production melt.[3] Depending upon the size and number of pieces the riser is broken into, it may be compactly stored on the bottom of the can or have the bulk of the material positioned rather high in the can depending on stacking by the operator. Figure 14 shows results of the Shuffler riser measurements. Most items were assayed with the material at the bottom of the can and then the material was redistributed so as to have the center of gravity as high as possible. Data from the two stacking modes are indicated in Fig. 14.

Data plotted in Fig. 14 indicated that either the two stacking modes yield about the same result, or the item with the higher center of gravity had a considerably lower assay result. Two effects tend to make assays of high center of gravity items low. First, material in the upper half of the assay chamber yields a lower delayed-neutron response because the material is farther from the

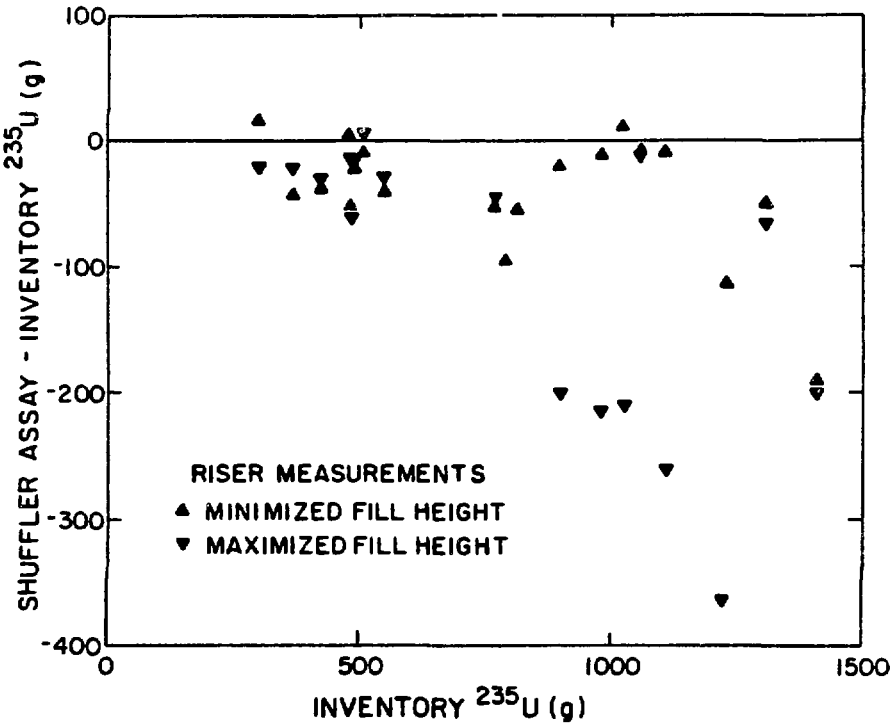


Fig. 14. Difference between the inventory ^{235}U content and the NDA measurement made by the Shuffler for risers (Los Alamos Neg. No. 80-7656).

bottom detector bank resulting in a decreased counting efficiency. The second effect is that the self-shielding correction over corrects because the material is misinterpreted to be more spread out than it actually is. If material is stacked so as to have the response dominated by chunks in the upper half of the scrap can, then the nonuniform calibration is used to analyze the data, and the assay value is in better agreement with the assay from the material at the bottom of the can.

Because of the heterogeneous nature of this material, strict administrative control is necessary to obtain reliable assays. Broken risers should be stored as compactly as practical in the scrap cans, and the fill height should not exceed half the can height. If necessary, two scrap cans could be used to store the pieces from a single riser. An alternative approach would be to to cast riser material in DR ingot molds prior to assay. This procedure has the advantage of a more accurate and reliable assay, but the disadvantage of adding a step to the recycling operation.

4. Cores. Technically, cores are not part of the scrap and waste materials; however, core measurements offer an opportunity to evaluate the Shuffler technique for an additional application. Cores are an intermediate step in the fuel tube manufacturing process. The cores have already undergone machining, cladding, out-gassing, and extrusion. An additional extrusion with aluminum-enclosed cores is used to produce fuel tubes.[2]

Figure 15 shows results from core assays. Data clustered vertically at about 700 g ^{235}U are from inner cores, and data at about 1400 g are from middle cores. The remaining items include cores produced for Shuffler testing, those fabricated for evaluation of the Random Driver, and other special items.[36] Data for the inner and middle cores differ by slightly more than expected from combined uncertainties in the Shuffler assay and inventory values. The difference

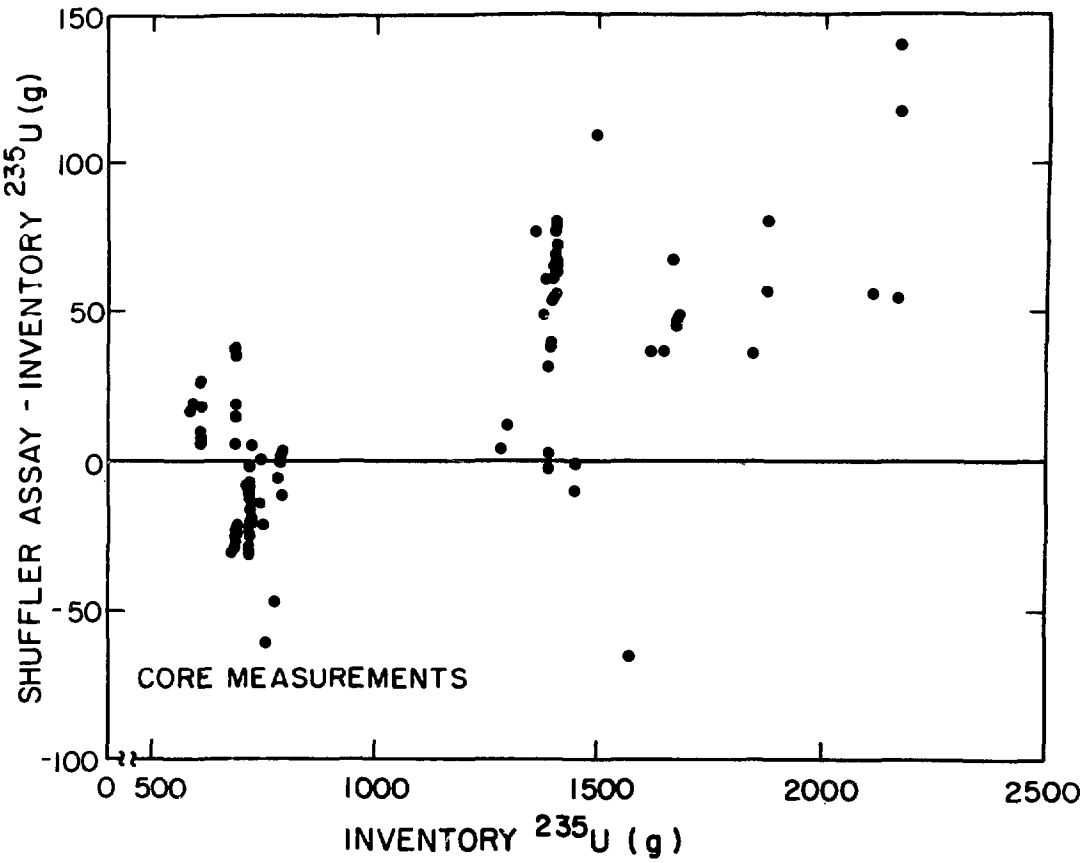


Fig. 15. Difference between the inventory ^{235}U content and the NDA measurement made by the Shuffler for cores (Los Alamos Neg. No. 80-7655).

might be attributable to the cores being hollow and the Shuffler being calibrated with solid standards. However, the spread in the data is also larger than could be reasonably expected from statistical fluctuation. Observed fluctuations are about 2%, and the expected variation is less than 1%. Variability in the geometry is not a factor because cores are finished to the same size for each core type. Thus, we are forced to accept that the variation in the assay results is attributable to an actual difference in the ^{235}U content. Nevertheless, the results could still be biased in terms of the total ^{235}U content.

Figure 16 shows the average response for cores cut from a single extruded log. If the alloy were uniform throughout the log, the assayed ^{235}U should be proportional to the core weight, and the response would be flat within statistics along the length of the log. However, the data in Fig. 16 indicate that the relative ^{235}U content varies by about ±1.5%. The numbering corresponds to their respective positions in the log from which they were cut with the low numbers being the log end that was first through the extrusion press. The lower ^{235}U content in the first section in the extrusion process is interpreted as being caused by the aluminum cladding extruding more easily, and thus, more aluminum and less uranium alloy is present in the first part of the extruded log. As the extrusion continues, the alloy catches up causing the high value in core section 3; and thereafter, the extrusion is more uniform as equilibrium conditions are approached. Data obtained by Shuffler confirmed some theoretical details of the extrusion process and provided measurements of the magnitude of various effects.

5. Fluxes. Two types of flux materials indicated as LF and LX in Fig. 1 are assayed by the Shuffler. Both these materials contain uranium in small quantities and low purity. As noted in Fig. 1, LF ingots are screened for uranium by a gamma-ray detector before the material is sent to the burial ground. If an LF ingot contains sufficient uranium, it is returned to the leach run for further recovery.[3] Until recently, LX material was also returned to a leach run; however, evaluation of this low-purity scrap recovery operation suggested that recovery could be more efficiently performed at Oak Ridge using a different process. Procedures for this option are being developed. Accountancy for these shipments will be made by measuring LX material prior to its shipment to Oak Ridge.

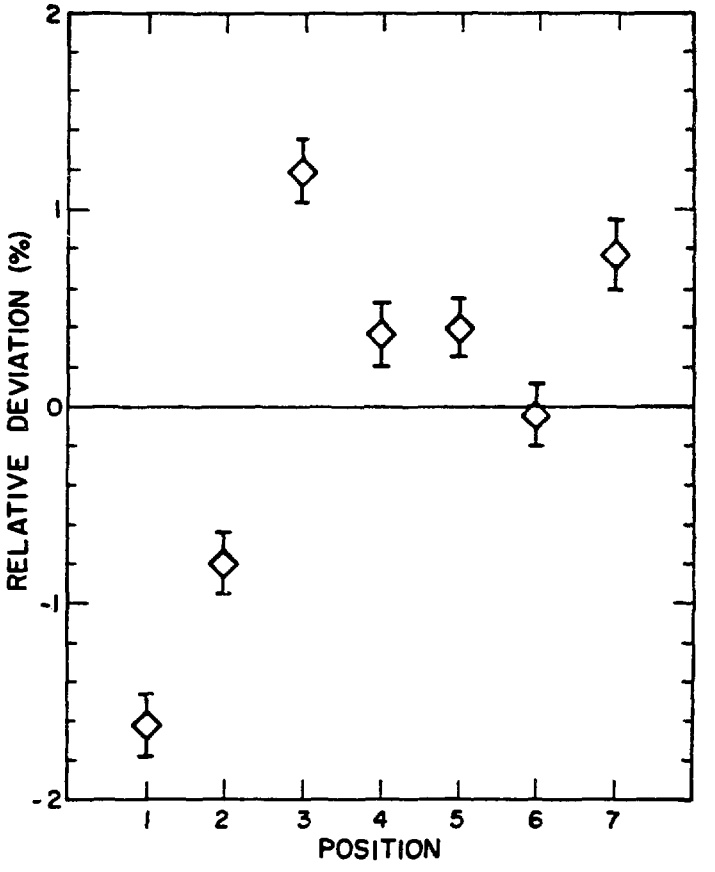


Fig. 16. Relative variation of the ^{235}U content as a function of the core position after the first extrusion (Los Alamos Neg. No. 80-7657).

As was mentioned in Sec. IV. D., producing standards for leached material is not practical because the uranium alloy separates during the manufacturing process. Instead, standard lathe chips were added to flux material to produce a calibration curve for this material type.[37] Figure 17 shows the calibration data. The data fit well to a linear calibration. Linear extrapolation estimates that the initial ^{235}U content was 12.2 ± 2.1 g. The Shuffler calibration employing correction factors estimated the ^{235}U content at 12.8 ± 0.7 g. Thus, the general calibration with correction factors appears to be adequate for flux material measurements. In order to further check the accuracy of the general calibration, two additional tests were made. First, assay results made with the general calibration were compared to the specialized calibration made for

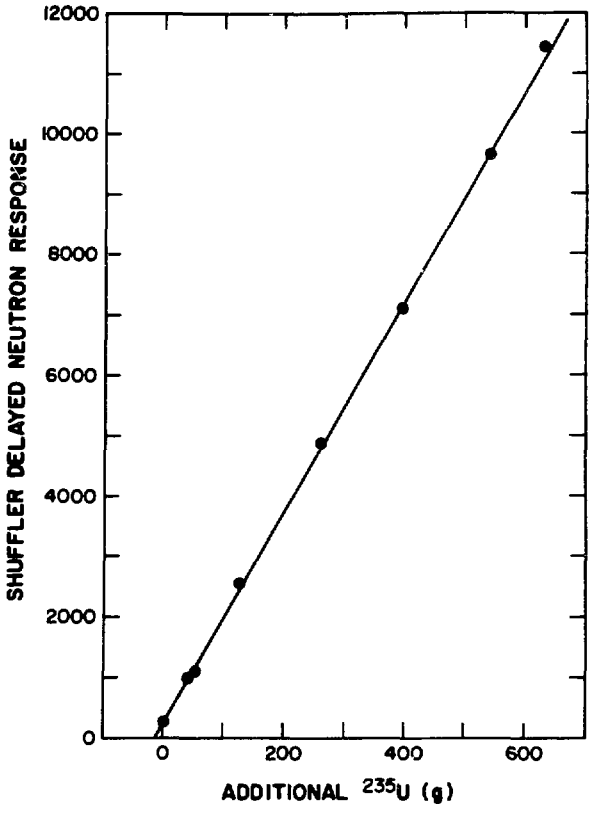


Fig. 17. Test measurements made for LF flux by successive additions of standard uranium-aluminum alloy lathe chips (Los Alamos Neg. No. 80-9577).

flux material. Second, LF flux assays were compared to results from a NaI-based pulse height analysis (PHA) system. Agreement between the general and specialized calibration was excellent. Because no advantage could be gained and multiple calibrations are a distinct disadvantage over a single calibration, the generalized calibration is recommended for flux material.

Comparison of the Shuffler assay results with the NaI PHA system LF ingot results proved interesting because the results were not the same. The gamma-ray assay was consistently about a factor of two higher than the Shuffler result. Because of the very detailed evaluation of the Shuffler at SRP and the expected behavior based on Monte Carlo transport calculations, it was considered unlikely that the gamma-ray based assays were correct, and the Shuffler result was low just for this material category. Further investigation of the gamma-ray system traced the source of the discrepancy to a calibration error. It should be noted

that the gamma-ray system was used for waste screening, and thus, more conservative restrictions were enforced because of the high assays. The Shuffler will replace the gamma-ray assay, but the PHA system will be retained as a backup to the Shuffler.

6. Floor Sweepings. As the name implies, floor sweepings are materials collected from process area floors. This category has the greatest material type variation. The uranium comes from spills in the casting area, chips not caught by screens, and bits and pieces such as from breaking of risers for storage in scrap cans. Other materials in floor sweepings include graphite, paper, and dirt. The dirt originates more from settling dust than from pieces of soil, because shoe covers worn in the process area reduce the amount of dirt tracked in as well as the amount of contaminates tracked out.

As indicated in Fig. 1, floor sweepings are not processed at SRP but are shipped to Oak Ridge for recovery. Floor sweepings are collected in scrap cans, repackaged in one-pound coffee cans for assay, and then transferred to No. 10 size cans in order to meet shipping requirements. The NaI-based gamma-ray detection system assays the material in coffee cans because the smaller diameter reduces gamma-ray shielding and thereby improves the measurement accuracy. Nevertheless, self-shielding associated with lumps is still present in smaller cans.[38] The uncertainty assigned to the assay is ±30%.[39]

The Shuffler measured floor sweepings in scrap cans and No. 10 cans. A comparison was made of the assay results for scrap can measurements and the sum of the No. 10 can content's measurements. The average bias was about 4% with the scrap can measurement indicating the higher ^{235}U content. The largest discrepancy was 6%, and the indicated accuracy of the Shuffler results ranged from 1 to 2%.

Figure 18 shows a comparison of floor sweeping material assays packaged in No. 10 cans with the PHA measurements. The data are within the conservative ±30% errors assigned to the PHA measurements. The errors bars are those of the Shuffler measurements. The agreement between the two instruments is quite good for items containing up to about 140-g ^{235}U. Above 140-g ^{235}U, the PHA system tends to give lower assays, undoubtedly due to the increased possibility for self-shielding with larger quantities of uranium.

The heterogeneous nature of floor sweepings allows the possibility of having the bulk of the uranium in the upper half of the can. About 2 to 3% of

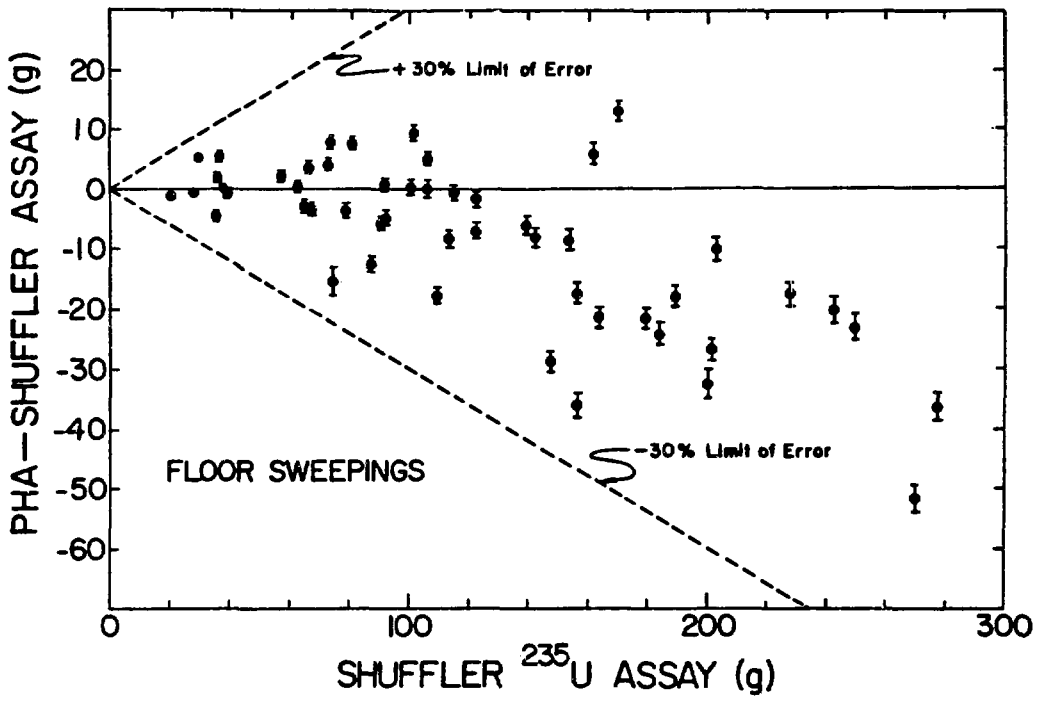


Fig. 18. Comparison of the Shuffler and the SRP NaI-based pulse height analysis (PHA) system for floor sweepings (Los Alamos Neg. No. 80-8068).

the floor sweeping cans result in the message prompting the operator to repackage the material into two cans to improve the assay accuracy. In one case, the can that led to the message was inspected and found to contain pieces of alloy scraped from graphite molds on top of usual floor sweeping material. In another case, when the operator received the message, he dumped the contents into another can, thus reversing the order of the contents in the can. The material was then assayed without generating the warning message. Thus, for floor sweepings the discretion of the operator could be used to determine the best course in terms of either dividing, segregating, or redistributing the can contents that do not initially meet the uniformity criteria.

Establishing an overall accuracy for this material type is difficult because of the wide variation in composition, and no alternative technique exists that can reliably measure all items. The gamma-ray technique fares reasonably well for homogeneous items, and accuracy in the range of 1 to 3% with trans-

mission corrected scans using high resolution Ge(Li) detectors appears possible.[19,40] However, lumping that results in self-shielding is a limitation. An alternative scheme for estimating the Shuffler accuracy would be comparison with recovery data. A drawback, in addition to the long time required for recovery, is ambiguity caused by unrecovered material remaining in waste residues or left in process lines. Nevertheless, long-term data would be useful for identifying trends. Thus, accuracy estimations have been drawn on results of special case assays where known quantities of ^{235}U are added or when the waste is redistributed within the can. In the latter case, checks built into the assay limit the acceptance of data from extreme situations for both normal assays and test measurements. In addition, experience with other materials, such as the deliberately nonuniform combination of pure aluminum and U-Al disks, aids in estimating the limits of error. Thus, the floor sweeping accuracy is estimated at 1 to 12% with the larger errors being caused by nonuniformly mixed items having the predominate uranium content within 2 to 5 cm from the base of the can. Because of the sensitivity at this fill height, the Shuffler assigns larger uncertainty limits, as can be noted from Item 12 in Table V. Because of the heterogeneous nature of this material category, items with high ^{235}U content (>500-g ^{235}U) can have large inventory errors. Thus, floor sweeping items with more than 500-g ^{235}U indicated by the Shuffler assay should be inspected for uniformity, and the possibility of process material being included with floor sweepings should not be discounted.

VII. SUMMARY

During the test and evaluation at SRP, the ^{252}Cf Shuffler first underwent operation and safety tests by the Equipment Engineering Department (EED) and later measurement accuracy tests by the Reactor and Reactor Materials Technology Department (RRMTD). Operational and safety recommendations made by EED were fully implemented prior to installation in the reactor fuel fabrication facility.[11] At the fuel fabrication facility, RRMTD personnel have been responsible for evaluating the Shuffler and fabricating standards and materials needed for testing performance for various scrap and waste categories. In addition, measurements were made with some process line materials.

Equipment failures have occurred with the Shuffler during its evaluation at the fuel fabrication facility, but they have been minimal. Most repairs are made by module exchanges, and actual repair is done off line while the Shuffler continues to operate. The most serious failures occurred with the load cells, which require several hours of labor because access to the interior of the Shuffler is necessary.

The long-term stability of the instrument has been good. Assays of certain items indicate that the detectable drift is less than 1% over the 15-month evaluation by RRMTD. To reduce the drift, corrections for the most likely causes have been implemented, and the stability will continue to be monitored as part of the routine operation of the Shuffler. A recalibration procedure has been suggested to further reduce the drift if it continues.

Because of the diversity of scrap and waste materials, the most direct method would be to have a separate calibration for each material category. Disadvantages of this approach are that a set of standards for each material type is needed (especially difficult for low-purity items), and safeguards and quality assurance problems could occur if an item were assayed using the wrong calibration curve. Instead, a single calibration with correction factors based upon auxiliary measurements made during the assay was devised. The correction factors account for neutronic effects such as self-shielding, multiplication, or moderation to relate responses from various material categories to the equivalent response of high-purity items. The advantage of the single calibration curve is that only standards exhibiting the neutronic effects are necessary, and assay of the material categories is accomplished by linear interpolation or extrapolation of correction factors. In practice, it was found that standards produced for high-purity DR ingots and for saw and lathe chips provided adequate material for calibrating the Shuffler for the full range of scrap and waste at the fuel fabrication facility.

The Shuffler accuracy depends upon the precision of the technique, the accuracy to which the standards are determined, and matching standards to the material being assayed. By comparing analytical results from different techniques at various laboratories to determine the ^{235}U content of an item based upon sampling, the achievable accuracy is about 0.5%.[20] Because of the steps involved in sampling, analyzing, and interpreting results, significant improvements for the Shuffler standards are not imminently expected. Precision of the

Shuffler is better than 0.1% for uniform materials. However, for heterogeneous loose materials, variations up to 12% are possible before the nonuniformity is detected by the Shuffler. Matching standards to process materials is done by first choosing standards that are made like process items except that the material is directly traceable to well-characterized feed material, and sampling is done to further assure accuracy.

Accuracy on a material-by-material basis generally becomes worse for low-purity or poorly characterized categories. For DR ingots, the accuracy is about 0.5 to 2%. For chips, which have a greater variability because of their different shapes, sizes, and compressibility, the accuracy is about 1 to 3%. Risers are a category that needs strict administrative control because pieces may be stacked more or less compactly. In an uncontrolled situation, errors up to 30% are possible. If risers are melted and cast in DR ingot molds, accuracies of 0.5 to 2% are expected. For flux material, accuracies of 1 to 4% would be expected. Floor sweepings can expect an accuracy of 1 to 12% with the typical accuracy being about 3%.

When the uranium isotopic mixture blended at SRP changes from the current value of 60% to about 50% ^{235}U, assay values given by the Shuffler will be about 1% high. The situation can be corrected by fabricating a new set of calibration standards at 50% enrichment or perhaps lower to give a longer useful life for the new standards as the enrichment is eventually further reduced. A second approach would be to precisely determine the shift in the assay value with a few standards produced at the new enrichment. Then after measuring the bias between the existing 60% enrichment standards and the new material, one simply changes the ^{235}U values assigned to the current calibration standards to values that give the correct ^{235}U assay when items of a different enrichment are measured. This second approach could be used as a stop-gap measure until a full set of standards is produced at the new enrichment.

Accuracy, safety, reliability, and ease of use of the Shuffler have been evaluated at SRP to determine the practicality of the instrument's routine use in an industrial process facility. The Shuffler was upgraded to meet strict safety requirements of an E. I. du Pont de Nemours and Company operated facility. The instrument has been as reliable as others of its complexity, and field repairs are expedited by the inventory of spare parts. The ease of use is exemplified by an operator who was introduced to the unit at the start of the day and then demonstrated it to outside visitors later the same morning. In

terms of accuracy, the Shuffler equals or exceeds the accuracy of techniques currently being used to measure the ^{235}U content of scrap and waste material at the fuel fabrication facility. In addition, the Shuffler can measure items that were previously unmeasured, and it eliminates sampling errors by measuring the entire item. NDA measurements made by the Shuffler require between 8 and 20 min while sampling and chemical analysis takes about 2 weeks. In spite of advantages offered by the NDA measurement, it cannot replace chemical analysis because the uranium isotopic data is needed for items in a production melt. Comparison of chemistry results with those of the Shuffler can catch erroneous results that might otherwise go undetected. In summary, the ^{252}Cf Shuffler met the requirement of improving the accuracy and timeliness of safeguards and accountability. A list of additional documentation describing the Shuffler and the Test and Evaluation Program is found in the Appendix.

ACKNOWLEDGMENTS

Development and evaluation of the ^{252}Cf Shuffler extended over a three-year period during which many people contributed their expertise to make the program a success. First, I would like to thank the following Los Alamos personnel and acknowledge their contributions. S. E. Beach handled the liaison between Los Alamos and SRP. S. C. Bourret led the electronics design and selected the computer and peripheral components. R. L. Brewer helped with the assembly and californium source attachment. P. R. Collinsworth participated in the detector electronics design and made the final assembly and tests of the neutron detector system. L. R. Cowder directed the californium source attachment. H. R. Dye contributed to mechanical fabrication and assembly. G. W. Eccleston was consulted on neutronic calculations, wrote many of the software drivers, and participated in initial testing at Los Alamos. E. A. Gallegos helped with electronics design, fabrication, and testing. D. C. Garcia participated in mechanical fabrication and assembly and directed transportation and set-up of the Shuffler at SRP. D. L. Garcia made improvements in the electronic modules and verified the completeness of schematic drawings. S. S. Johnson wrote the disk-handling software and helped locate software bugs. K. E. Kroncke did the mechanical design and helped with assembly. M. M. Meier and C. J. Nachtsheim reviewed the ^{235}U mass assignments for the calibration disk standards. H. O. Menlove was consulted on design and operation features.

G. G. Ortiz helped with assembly. D. L. Peterson helped with electronic module layout and fabrication. C. O. Shonrock helped with mechanical assembly. L. G. Speir directed the mechanical design and assembly. C. A. Spirio, Assistant Safeguards Technology and Training Group Leader, provided direction for technical support personnel. J. E. Swansen led the neutron detector preamplifier design. G. Walton contributed to drafting. And, R. B. Walton, Safeguards Technology and Training Group Leader, was responsible for the overall Los Alamos effort.

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APPENDIX

CALIFORNIUM-252 SHUFFLER DOCUMENTATION SUMMARY

In addition to this report, the documentation for the SRP ^{252}Cf Shuffler includes formal presentations published in conference proceedings, informal reports issued by both Los Alamos and SRP, contibutions to Los Alamos and SRP progress reports, photographs, and mechanical component and electronic schematic drawings. The above information is available upon request; a list of the documents follows.

Conference Proceedings Publications

T. W. Crane, S. Bourret, G. W. Eccleston, H. O. Menlove, L. G. Speir, and R. V. Studley, "Design of a ^{252}Cf Neutron Assay System for Evaluation at the Savannah River Plant Fuel Fabrication Facility," in Analytical Methods for Safeguards and Accountability Measurements of Special Nuclear Materials H. T. Yolken and J. E. Bullard, Eds., (NBS Special Publication 528, U.S. Government Printing Office, Washington, D.C., November 1978), pp. 41-48.

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Drawings (Available from the Technical Information Center, Oak Ridge, Tennessee)

Electronic Boards	68Y-155585- D-57 through D-62
Six-Channel Scaler	4Y-223041 R3, C1, and C2
Mechanical Assembly	68Y-155522 D-1 through D-61

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