

NATIONAL BUREAU OF STANDARDS REPORT

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INTEGRATED SAFEGUARDS EXPERIMENT CAMPAIGN 1 REPORT

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U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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Abstract

Details of the planning, conduct, and results of Campaign 1 of the Integrated Safeguards Experiment established at the General Electric Company, Vallecitos Nuclear Center, Pleasanton, California, are reported. A material balance based on completely measured values, for materials containing significant amounts of Plutonium, was calculated. The impact of the use of nondestructive measurement equipment and methods was studied. Conclusions drawn from the results of Campaign 1 and related suggestions for refinements to procedures for Campaign 2 are made.

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Overview

The nuclear materials safeguards program to guard against the loss or unauthorized diversion of nuclear materials has long been hampered by the lack of material balances based entirely on quantitative measurements. The results of the Technical Studies program in the development of non-destructive assay instruments have made possible the timely measurement of many hard-to-measure materials, leading to the possibility of completely measured material balances.

The Integrated Safeguards Experiment (ISE) was established at the General Electric (GE) Vallecitos Nuclear Center (VNC) with the broad purpose of testing the usefulness, to Safeguards, of materials balance accounting techniques which employ data based completely on quantitative measurement. The concept is that, if all streams and outlets of fissile product are accurately determined and the uncertainties associated with each measurement accounted for, then such material balances would provide control over nuclear materials within a production facility with a known precision. If such balances can be made often enough, and if the balances are precise enough, then to what extent might this satisfy the requirements of the Domestic Safeguards Program and specifically for plutonium fuel processing plants? It

is in this sense that the word "usefulness" is intended. To provide a basis for comparison with previous GE practice in materials accounting, the company made historical data, compiled from a GE campaign conducted prior to Campaign 1 available to the ISE team. The evaluation of these data is not shown in this report. The data may be satisfactory for routine material control for GE, but do not permit the calculation of an accurate and meaningful material balance satisfactory for safeguards use. The ISE was planned to be run in several campaigns, with changes and refinements in procedures to be added to each succeeding campaign as dictated by experience in the previous one. Campaign 1 followed a plan carefully developed by GE and approved by the AEC and other participating ISE representatives. In addition, the Integrated Safeguards Experiment team followed details of the work very closely and thus were able to detect deficiencies in the procedures, as well as error sources which would require corrective action to insure more accurate material balances.

The experiment demonstrated that completely measured material balances are possible on a routine basis through the use of non-destructive measurement techniques coupled with the usual wet chemistry analysis. The addition of non-destructive testing techniques (NDT) to measure every known stream in the fuel fabrication line was an innovation

in a production environment which was previously considered in the R&D stage. Non-destructive testing has had very little use by commercial industry for quantitatively determining plutonium content in any type material.

Routine manufacturing practices were followed by GE with the exception of the use of NDT for measuring the Pu content of feed and special samples, as well as for scrap and waste materials.

The material unaccounted for (MUF) was calculated to be 112 gPu with a limit of error (LE)* of 198 gPu. The MUF value represents 2.4 percent of the Pu throughput (defined as feed input minus feed remaining in the inventory). The LEMUF was due almost entirely to the LE in the calorimetry-determined feed material. The major LE of the calorimetry measurements was due to the uncertainty associated with the determination of Pu238 and Am241 content of the material. Using isotopic measurement capabilities provided by New Brunswick Laboratory, the LEMUF would be reduced to 36 gPu, which indicates a MUF statistically significantly greater than zero. The MUF value for Campaign 1 would then be considered exceptionally high compared to LEMUF, especially since great effort was made to account for all material flowing through the process line during this campaign.

*The term "limit of error" (LE), throughout this document, refers to the range within which the value will fall with 95% confidence, assuming a normal (Gaussian) distribution.

One primary objective was not realized, namely that of determining process loss by unit operation. This objective was aimed at localizing process losses and possibly developing procedures to reduce such losses. This objective was not realized because of a moisture absorption problem which changed Pu concentration values up to 0.5%. This condition created LEMUF values for the unit processes which were too large when compared to the expected MUF values thus obscuring process loss information.

The data acquisition system, designed by GE to demonstrate material balance calculation and timely and perpetual inventory reporting, did not materialize during the term of this campaign. This feature, which hopefully will be implemented later, will be an important adjunct to a material control system.

The GE-VNC personnel responsible for calculating material balances were not experienced at data analysis and the statistical treatment of NDT measurement data, which were particularly complicated. If this is typical of the situation throughout the Industry, it may be necessary to engage in extensive education and training programs for personnel who will be responsible for this work.

Prior to this experiment, the effective limits of accuracy were established by product specifications,

which were reported to be ± 5 percent. Measurement capability was therefore secondary, particularly with respect to its application for safeguards purposes. The net result of this reasoning was to reduce the importance of accurate and timely measurements. For example, results from the chemistry laboratory were, in some cases, nearly a month in forthcoming after receipt of samples from the production line.

The benefits which will accrue to the company and to the Safeguards Program by making possible accurate and timely determinations of plutonium of heretofore difficult-to-measure materials are obvious. The above was not accomplished in Campaign 1 due to several reasons, as discussed in this report. It is clear that much education and training will be needed before ND measurement techniques may be used effectively.

The conduct of Campaign 1 indicates a strong need for standardization of methods of calibrating measuring equipment, sampling methods, and minimum reporting requirements.

Report On

Campaign 1 of the Integrated Safeguards Experiment

I. Introduction and Background

In January 1968, the USAEC, Office of Safeguards and Materials Management established a Safeguards System Studies program which had as a primary objective the study and analysis of the various nuclear fuel manufacturing processes, for the purpose of developing a basis upon which criteria for acceptable limits on material unaccounted for (MUF), normal operating losses (NOL), shipper-receiver (S-R) differences and unmeasured special nuclear material (SNM) inventory could be established. Necessary information was to be compiled from both historical and current plant files and from it develop analytical models which could be used to evaluate plant data leading to the establishment of criteria for acceptable limits for MUF, NOL, S-R differences and other critical factors. Visits to licensee plants soon established the fact that historical data was lacking in quality and depth and therefore not applicable to the establishment of criteria to be used in licensee plants. Also current data was not applicable since much of it was based on questionable measurement data.

As a result of the findings of this early industry survey the AEC decided to conduct Integrated Safeguards Experiments (ISE) within licensee plants which were producing nuclear fuel materials of major critical interest from a need-to-safeguard viewpoint. The purpose of the Integrated Safeguards Experiment was to test the usefulness, to Safeguards, of materials balance accounting techniques which employ data based completely on quantitative measurements.

A. Objectives

The ISE was planned to accommodate four major objectives:

1. Evaluate the impact of recently developed non destructive (ND) measurement methods on materials balance accounting.
2. Study methods by which measurement data are acquired, processed and analyzed in the facility's nuclear material control system.
3. Evaluate the accuracy and timeliness of currently employed chemical analysis and sampling.
4. Determine the magnitude of unidentified process losses for specific unit processes and possible methods for reduction and isolation of these losses through use of submaterials balance areas.

A secondary objective was to determine the possibility of utilizing the results and experience from an ISE to formulate numerical criteria and standard procedures for an "across the board" use in AEC licensee plants. Since plutonium is currently the highest priority material for safeguards and since for at least the next decade, the major use of plutonium in the commercial power cycle will be utilization in thermal reactors, a conclusion was reached to conduct the first ISE in a plutonium fuel fabrication plant.

B. Contractor Selection

After evaluation of proposals submitted by several companies, The General Electric Company Plutonium Fuels Laboratory, Vallecitos Nuclear Center near Pleasanton, California was selected for the first ISE. The ISE was planned to cover several campaigns, each one comprising a complete fuel manufacturing cycle and each beyond the first containing additions brought about by experience gained from the previous campaign procedures. This report describes the first campaign completed at the GE Plutonium Laboratory.

C. The Integrated Safeguards Experiment Team

An ISE team comprising representatives from the USAEC (OSMM), Brookhaven National Laboratory (TSO) and the National Bureau of Standards (NBS-TAD) provided on-site representation during the conduct

Campaign 1. These ISE personnel, referred to as ISE Technical Representatives, worked jointly with GE personnel during the planning, experiment operation, and evaluation of data taken during the campaign.

D. Utilization of the AEC Plant Instrumentation Program (PIP)

To be useful to Safeguards, a materials balance account must contain quantitative measurement data on all inputs and outputs to the system. Previous to the ISE, dirty scrap, disposable waste, and plutonium content of finished fuel rods were among the materials that had not been measured. The utilization of a range of NDT methods was utilized as a means of diminishing the uncertainties in the quantities of starting materials, intermediate, scrap, product and inventories. It was expected that ND measurements would permit timely and less expensive determinations of amounts of materials and in certain material balance areas (MBA, permit closing of the material balance loop.

II. Description of Plant and Process Operations

A. Glove Box Operations

The Plutonium Fuel Laboratory is comprised of two sections as indicated on the floor plan, Figure 1. The equipment on the right half is primarily for fabrication of limited quantities of mixed oxide ceramic fuel for specific test purposes. In this area also are included facilities for material and property studies and auxiliary fuel fabrication operations. The equipment in the left half of the laboratory is primarily for fabrication of test and demonstration fuel for both fast and thermal reactors. It is in this half of the laboratory that the Integrated Safeguards Experiment was conducted. The work was done exclusively in a self-contained, interconnected glove box line. Each individual box contains two inlet and two outlet ports, each being covered with an absolute filter. In addition, the outlet port absolute filters are preceded by double thickness prefilters. Individual boxes are equipped as follows:

Box 37

V-Blender

Hammermill

Mettler P-5 Balance

Torsion DLT-2-1 Balance

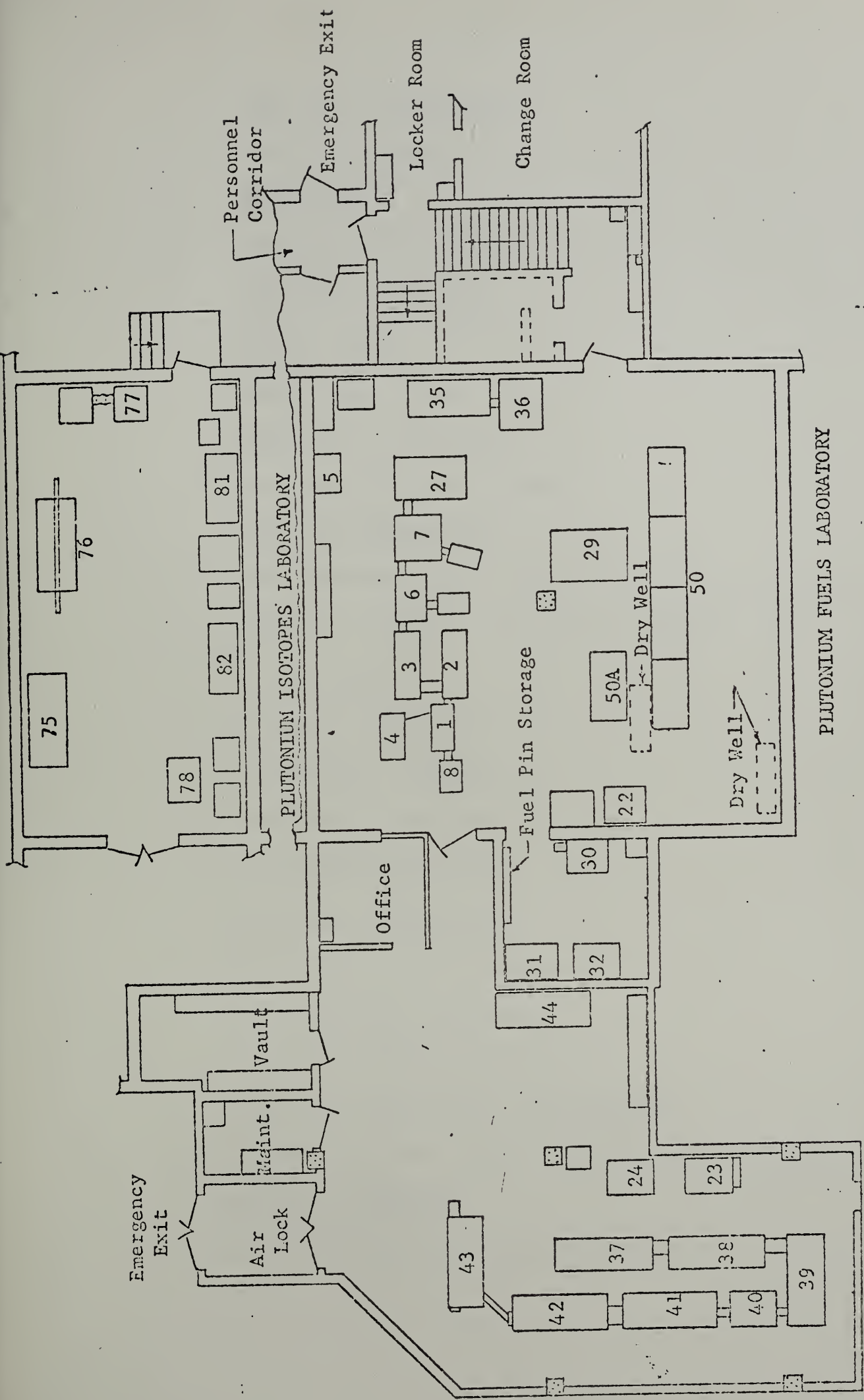


Figure 1: GE-VNC Plutonium Fuel Laboratory Floor Plan

Box 38 - Prepressing

Slugging press

Granulator

Mettler P-5 Balance

Box 39 - Pressing

Pellet Press

Small ball mill

Mettler P-5 Balance

Torsion DLT-2-1 balance

Box 40 - Sintering

Push through type furnace (total in-furnace time 12.5 hours - hot zone (1650°C) time 2 1/2 hours).

Box 41 - Grinding

Centerless grinder

Mettler P-5 Balance

Two vacuum cleaners

Box 42 - Inspection

Mettler P-5 Balance

Mettler 160-N Balance

Ainsworth Model 10 Balance

Dimension Measuring Instruments

Oxygen to metal (O/M) determination furnace

Box 43 - Loading

Cyclotron pellet feed mechanism

Mettler P-5 Balance

V-trough

Box 43A - Rod Decontamination

B. Flows and Material Movements

Material movement through the fabrication process is summarized as follows: Bulk weights of PuO_2 and UO_2 , as received in cans (PuO_2) and plastic bags (UO_2), were established prior to introduction to the process line. This weighing was done to establish conformance with the weight on the Shipper Transfer form. A separate sample of PuO_2 obtained from the feed cans was sent to the analytical laboratory for analysis.

Cans containing PuO_2 were measured by calorimetry prior to their use for batch make-up for this campaign.

Material flow was initiated with Box 37. Feed material was entered as PuO_2 and UO_2 (critical limit 2500 grams fissile.) Calculated amounts of PuO_2 and UO_2 were weighed out and about 8 kg. of material was blended. The output from the blender was split into four or more cans. Another blend was made and split out. This process was repeated until each can contained about 8 kg. The mixed oxide (MO_2) was then put through a hammermill, then back into the cans.

The contents of each can were then re-blended and weighed. Usually 3 of the cans were sampled, (1 to 10 grams per sample) and Pu content determined. Output from this box was cans of mixed oxide.

Material was then transferred to Box 38 where it was prepressed (slugging operation to densify material), granulated (to stabilize particle size) and weighed.

Slugging and granulating conditions the oxide powder for the pressing operation which took place on Box 39.

Granulated powder was mixed with a binder prior to pressing. Pressed pellets were loaded into tared molybdenum boats, gross weighed and put into the sintering furnace (Box 40).

After sintering, the loaded boats entered Box 41 where they were weighed and pellets checked for diameter. Oversize pellets were centerless ground and all pellets were then returned to the respective boats in which they were loaded as they were transferred to Box 41. Boats were again weighed and the difference in weight was assigned to grinder sludge.

Boats, loaded with finished pellets, were then transferred to Box 43 where 12% of the thermal-fuel pellets were inspected for weight, length and diameter. A pellet sample from every fourth boat was taken and transferred to the laboratory for chemical analysis. In addition to measuring pellets for Pu content, periodic checks were made for impurities, isotopic value and O/M ratio. Acceptable pellets were weighed into cans and transferred to Box 43 for loading.

Specific pellets were stacked and weighed on a tared V trough before loading into tubes. Tubes one end sealed, were partially inserted into Box 43, through Box 43A, for the loading operation. After each tube was loaded, the open end was decontaminated and temporarily sealed with a polyethylene plug. The loaded tube was then removed from the interconnected glove box line to Box 24 where a permanent plug was welded into place. Finished rods were then leak checked and removed to another laboratory area for additional inspection.

C. Points of Measurements - Routine Measurements

1. Equipment and Material Form

(a) Box 37 - Blending

- (1) Material - PuO₂ powder (90% fissile)
UO₂ powder (normal)
 - (2) Measurement equipment: Top Loading Mettler P-5 balance with precision ~50 mg.
 - (3) Procedure - Single weighing for UO₂; double weighing for PuO₂
- (b) Box 38 - Prepressing
- (1) Material - Mixed Oxide (PuO₂ + UO₂)
 - (2) Measurement equipment: Top Loading Mettler - P-5 balance with precision ~50 mg.
 - (3) Procedure - Single Weighing
- (c) Box 39 - Pressing
- (1) Material - Granulated Mixed Oxide plus Stearotex
 - (2) Measurement equipment: Top Loading Mettler P-5 balance with precision ~50 mg.
 - (3) Procedure - Single gross weighing of tared loaded boats.
- (d) Box 41 - Grinding
- (1) Type Material - Sintered pellets
 - (2) Measurement equipment - Top Loading; Mettler P-5 balance with precision ~50 mg.

(3) Procedure - Loaded boats are weighed before and after grinding.

(e) Box 42 - Inspection

(1) Material - Sintered pellets

(2) Measurement equipment: Top Loading, Mettler P-5 balance with precision ~ 50 mg. Micrometer for pellet diameter and length measurement; tapered calibrated rod for inside diameter and length measurement; tapered calibrated rod for inside diameter of annular pellets.

(f) Box 43 - Loading

(1) Material - Specification pellets

(2) Measurement equipment: Top Loading Mettler P-5 balance with precision ~ 50 mg.

(g) Other Measurement Methods Utilized by GE

(1) Controlled Potential Coulometer

This method is based on the quantitative oxidation or reduction of the element in question, under precisely controlled conditions. The number of coulombs required to do the oxidation or reduction is a

measure of the number of electro-chemical equivalents present of plutonium. The LE for a single determination of Pu content of sintered mixed oxide was calculated to be 0.6 percent.

(2) Isotopic Measurements

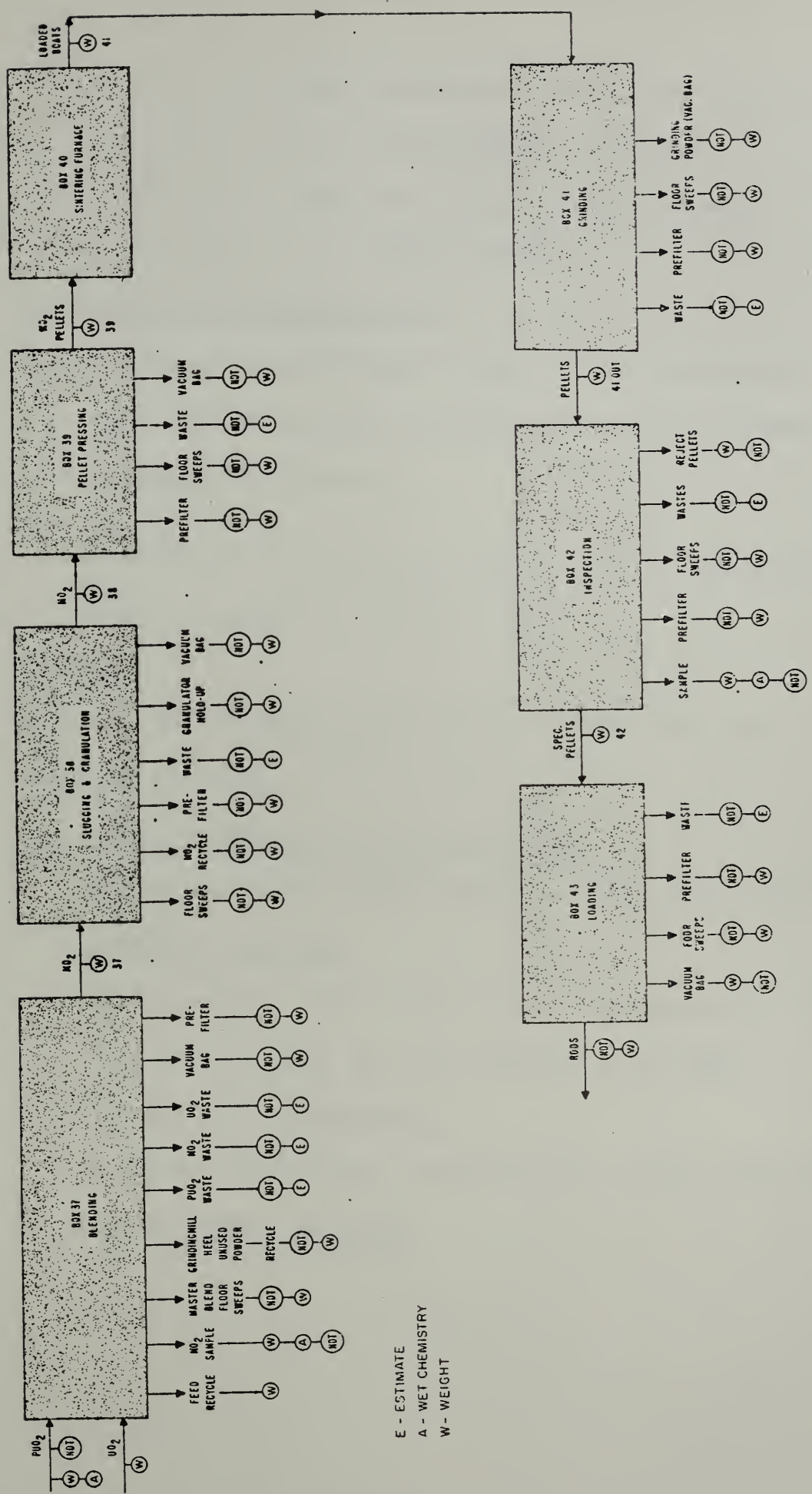
(a) Mass spectrometric measurements were used for Pu239, Pu240, Pu241 and Pu242.

(b) Vendors values were used for Am241 and Pu238. (alpha counting was used for Pu238 in campaign 2)

(3) NDT Measurements

(a) Calorimeter

This method relies on the principle of measuring the heat produced by the alpha decay of the various plutonium isotopes present. Knowing the concentration of each isotope in the material and the heat output per gram of each, the total Pu content can be calculated.



E - ESTIMATE
 A - WET CHEMISTRY
 W - WEIGHT

FIGURE 2 - POINTS AND TYPES OF MEASUREMENTS

(b) Spontaneous fission neutron coincidence counter.

(c) Germanium lithium gamma spectrometer.

2. Points of Measurement

Points of measurement, type of measurements made and materials measured are shown schematically on Figure 2.

A summary of individual measurement determinations is shown on Table II-A.

D. Records

Records maintained by GE during this campaign were as follows:

1. Process operating instructions

(Prepared and issued before start-up of campaign)

2. Transfer records

(Criticality Limit Area Inventory Sheets). Record all of box to box transfers of SNM.

3. Weight gain experiment

(Moisture adsorption observed for feed material).

4. Batch makeup records

TABLE II-A

	<u>Chemical Assay</u>	<u>Calorimetry</u>	<u>Neutron Counting</u>	<u>Gamma Scanning</u>
PuO ² Feed	7	13	-	-
Preproduction Samples	12	-	-	-
Production Samples	51	25	-	-
Scrap & Recycle	-	-	51	-
Discards	-	-	72	20
Completed Rods	-	-	-	28 (b)
Special Samples	26 (a)	-	40	-

(a) Performed by New Brunswick Laboratory

(b) Not completed

6. Processing Records

Records of the weight of material accumulated in each vacuum cleaner bag and filter assemblies in the processing line.

7. Cleanout Summary

As inventory of the Special Nuclear Material remaining in the glove box line after the completion of processing.

8. Losses

Sintering and grinding losses.

9. Analytical results

The analytical results for plutonium content (Pu/MO₂, O/M, plutonium isotopic and impurities in each master blend).

10. Standardization data

Data for plutonium, uranium, iron, and oxygen to metal ratio standardizations as well as information on daily balance variation.

E. Campaign 1 Feed Material

Plutonium dioxide feed material used in this campaign came from the same lot that was used in previous work by GE. It was noted, after examining historical data provided by GE for previous work, that Pu values submitted by the shipper were used in calculating material balances. As part of the ISE an attempt was made to obtain measured values

for this feed material. Such values would have to be provided not just for the ISE contract, but routinely, if safeguards for plutonium fuel fabrication is to be accomplished by materials balance accounting. Because of the concern of the Integrated Safeguards Experiment with the feed measurement as well as the requirement that the ISE campaigns generate material balances completely closed by measurement, chemical analyses were made on four of the six cans of feed material. Samples taken for analysis were found to pick up moisture from the surrounding atmosphere. This condition introduced a serious problem in determining the plutonium content of the feed. For more details of the moisture problem refer to Section IV B of this report.

F. PIP Instrumentation Available for GE-ISE Campaign 1

1. Passive gamma-ray instrumentation for measuring rods and low-level waste. This consisted of a 50 cm³ GeLi high resolution detector, several NaI(Tl) scintillation counters, amplifiers, single and multichannel analyzers.

(a) Utilization and observations

1. Passive gamma measurements. High resolution spectra of all the GE material to identify the gamma-ray peaks of interest.
2. Scan rods with GeLi. For mixed oxide rods containing 2-4% PuO₂, the counting rates are too low to compare content of individual pellets. The integral count for a whole rod gives adequate precision for safeguards measurement of total Pu.
3. NaI(Tl) gives high enough counting rate to be useful for quality control. GE devoted considerable effort to a crude (four bin) spectral analysis of the NaI data in order to measure the principle isotopes separately (Pu239, 241; Am241; U235, 238). Accuracy achieved to date is 2-5% for the Pu content using this

method. If one were to measure a set of rods of a given content against a similar standard the 2-sigma accuracy for the set should be well under 1%.

4. Both GeLi and NaI(Tl) measurements have been made of low level waste discards and filters. Variations in geometry and matrix degrade the accuracy of such measurements. Since, however, only a small fraction of the product stream ends up in these items, accuracies of 10-20% are acceptable, especially if the bias component is low.

2. Passive neutron instrument for measuring PuO_2 feed material, hard scrap and pellets. One 8" diameter by 10" high well counter coincidence circuit and scalers to record total neutron flux and coincident neutrons due to spontaneous fission.

The instrument provided by OSMM and designed by TSO consists of a polyethylene moderator 20" diameter, 24" high with an 8" diameter axial hole. The 24 BF_3 detectors, each 2" diameter by 20" active length are distributed within the moderating cylinder and operated in parallel. The efficiency is 8 to 18% per

neutron, depending on whether neutron absorbing sheets are inserted or withdrawn. The total neutron and the coincidence counting rates are recorded. Computations are required to make dead-time corrections corrections and to compute variances.

The instrument has been used to measure 1 kg cans of PuO₂ feed material, 1 gram to 2 or more kg of hard scrap and individual pellets containing 0.2 gPu. Calculations suggest that a precision of about 1% should be obtainable in a period of 5-20 minutes for samples containing 1 to 20 g of Pu240, longer times being required for larger and smaller samples. GE has shown that a precision of 1% can be obtained in two hours on one pellet containing about 30 mg Pu240, showing that the original calculations are pessimistic as regards the precision of coincidence counting of small samples. The method has proven to be useful for packages of dense scrap which have too much self absorption to be amenable to gamma analysis. Due to limitations in accuracy and errors which may be introduced by hydrogeneous materials in the matrix, this method has been viewed as primarily useful for quick checks

or verification. More efficient detectors could be made, for a price, and the results so far have been so encouraging that passive neutron measurements may play a more important role.

3. Calorimeters to measure PuO_2 and Pu-nitrate feed material, hard scrap and pellets. One large calorimeter and one small calorimeter using a common water bath with temperature control and sensing instrumentation.

One large calorimeter, suitable for measuring PuO_2 or $\text{Pu}(\text{NO}_3)_4$ (one liter bottles) feed material and one small calorimeter, together with water bath and controls, were borrowed from Mound Laboratory. The large calorimeter has given excellent results, with a precision of 0.025% reported for PuO_2 feed measurements and accuracy in the order of 0.5%. The smaller calorimeter does not have adequate sensitivity to measure individual pellets. A new pair of calorimeters are being built for the AEC by the Mound Laboratory for use in the ISE. The large one will be similar to the original. The smaller one will have higher sensitivity. The circuits will be designed to speed up measurements and to provide digital data.

III. Calibration and Measurements

The function of the calibration and measurement quality control program is to provide quantitative evidence on the operating characteristics of the measurement method and the conditions affecting the measurements.

The requirements for suitable calibration and measurement quality control programs were specified in Tasks 2A, 2B and 2F of the ISE contract. A separate task, Task 2G, was included in the ISE contract to provide a statement of work and funds for the systematic collection, storage and analysis of the data so obtained. Under Task 2G, a report summarizing the application and results of non-destructive and other measurement techniques will be prepared.

Historically, the calibration data obtained for the weight balances and wet chemistry methods were very informally treated. The limits of error presented by the plant did not reflect these data but were usually obtained from published reports. During Campaign 1, considerable time was spent with VNC personnel to develop methods for obtaining tangible evidence from the calibration effort which could be used in propagating the limits of error.

Table III-A shows the nondestructive measurements which were performed on Campaign 1 material. Reference standards were prepared for NDT by GE under the PIP (Plant Instrumentation Program) and were assayed along with the Campaign 1 materials.

Extensive calibration data were collected from which the measurement capability for the NDT instruments was determined. The LE on gPu in the PIP standards used in Campaign 1 are shown in Table IV-I of Section IV.

As a result of Campaign 1 experience a number of measurement and calibration problems were identified. The following is a brief description of these problems and possible solutions which were proposed.

Sampling errors (process variability) associated with Pu assay is perhaps the largest single component of measurement error in preparing the material balance. Consequently, limits of error propagated only on the basis of calibration error for the assay method underestimate the total error. The use of NDT methods, such as calorimetry, can be used to determine more accurately the size of the sampling error.

Standard measurement methods for assay of the plutonium dioxide and mixed Pu-U dioxide powders are noticeably affected by moisture content. The amount of moisture pick up is associated with the ambient humidity in the process line. As a result, a procedure for handling and drying of samples has been drafted. This procedure is given in Table III-B.

The weights of the exhaust filters obtained before and after the campaign were of limited use for estimating the amount of oxide hold up in the filters because of the loss of weight of those filters due to drying.

A procedure for equilibrating the weight of the exhaust filters was prepared, but was not used in this campaign. This procedure will be incorporated in Campaign 2. The procedure is based on the weighing and use of moisture equilibrated intake filters as exhaust filters.

Table III-A. Forms of Material

x - Denotes measurements available

	ND Technique				
	<u>Calorimetry</u>	<u>GeLi</u>	<u>NaI</u>	<u>Gross</u>	<u>Neutron Coincidence</u>
PuO ₂ Feed	x				
UO ₂ Feed					
PuO ₂ -UO ₂ Powder					
PuO ₂ -UO ₂ Pellets					
Fuel Column					
Fuel Rods		x	x		
Scrap				x	x
Recycle				x	x
Discards				x	x
Filters				x	
Replicate samples	x			x	x
NDT Specimens	x	x	x	x	x

Table III-B

Procedures for Sample Handling and Sample
Drying of Plutonium Bearing Oxide Powders

1. Pre-weight sample bottle (tare weight-1) and cap (tare-weight-2). Record all weighing data.
2. Immediately (within 1/2 hour) after the sample is drawn weigh the sample and bottle (gross weight-1).
3. If the sample is to be shipped offsite or stored onsite for more than several days the sample bottle should be hermetically sealed. If additional sealing tape or material) is added, gross weight-2 should be obtained.
4. After sample is subjected to periods of storage, check the gross weight.
5. Just prior to the analysis for plutonium, dry sample according to the following steps:
 - (a) Weigh the sample and bottle and check for agreement with gross weight. Record all data and report discrepancies.
 - (b) The sample is to be dried in the sample bottle. Open the sample bottle and obtain the sample and open bottle weight. Check to see that this weight plus the bottle cap weight equals the gross weight.
 - (c) Dry sample at 200 °C for 2 hours.
 - (d) After 2 hours, check for weight constancy every 15 minutes until weight change is negligible.
6. This procedure applies only to plutonium dioxide which was packaged and sealed in an atmosphere where the relative humidity is less than 5%. It does not apply in cases where the dry PuO₂ powder has been exposed to high relative humidity conditions and has picked up moisture. Also, there is no assurance that a sample, treated in this manner is representative of the feed material of oxalate (small amounts of which is normally found in PuO₂ produced from the nitrate form), is driven off in the drying process.

IV. Material Balance Analyses and Summary

A. Mixed Oxide Weight Balances

1. Discussion

The hood inventory sheets contain information concerning transfers of material into or out of each Criticality Limit Area (CLA). Each box is a CLA except for the pressing, sintering and grinding areas which are considered as one CLA. The weight balances that follow were computed on a gram of material basis from information appearing on the hood inventory records. The value listed under In represents the sum of all inputs.

2. Weight balances by master blend for each box.

Table IV-A shows box balances.

Table IV-B shows individual and cumulative summaries.

3. Observations

The following general statements were made upon examination of each weight balance:

- Box 37: The over-all weight gain in this glove box was a result of moisture and oxygen pickup during the master blend makeup operations.
- Box 38: A material gain with no attributable cause; most probable cause was either an erroneous or deleted transfer entry.

- Boxes 39, 40 and 41: The large weight loss was attributed to moisture, oxygen and volatiles which were driven off during sintering in a reducing atmosphere.
- Box 42 and Box 43; Individual weight balances indicated either a small material loss or gain. These variations appear to result from normal processing operations.
- A redundant entry was found when the transfer records were reexamined concerning Boxes 41 and 42.

A comparison between enrichments is more meaningful than between master blends within an enrichment. The reason for this is due to the cleanout procedures, which in the case of the master blends, permits the mixing of some of the first master blend scrap and waste materials with that from the second blend, thus somewhat confusing the issue. The situation is different between enrichments since a complete cleanout was made between enrichments.

TABLE IV-A Weight Balances by Master Blend for Each Box

<u>Material Identity</u>	<u>In</u>	<u>Out^(a)</u>	<u>Sample^(b)</u>	<u>Clean Scrap</u>	<u>Dirty Scrap</u>	<u>Waste</u>	<u>Oxide Balance</u>	
							<u>Blend</u>	<u>Enrichment</u>
<u>Box 37</u>								
M0094	32401.8	31791.2	3.0	—	401.8	15.6	190.2	—
M0095	32173.0	31905.6	3.0	236.8	215.3	20.3	-208.0	—
Enrichment 1	64574.8	63696.8	6.0	236.8	617.1	35.9	—	-17.8
M0096	32586.8	31881.4	5.0	—	604.8	2.3	93.3	—
M0097-8	42763.4	39995.5	11.3	1994.7	864.9	25.0	-128.0	—
Enrichment 2	75350.2	71876.9	16.3	1994.7	1469.7	27.3	—	-34.7
ISE Campaign 1	—	—	—	—	—	—	—	-52.5
<u>Box 38</u>								
M0094	32257.7	32608.8	—	—	—	14.9	-366.0	—
M0095	32408.6	32439.2	—	—	—	19.9	-50.5	—
Enrichment 1	64666.3	65048.0	—	—	—	34.8	—	-416.5
M0096	31882.4	31826.5	—	—	—	3.1	52.8	—
M0097-8	40133.5	40143.1	—	—	—	31.6	-41.2	—
Enrichment 2	72015.9	71969.6	—	—	—	34.7	—	11.6
ISE Campaign 1	—	—	—	—	—	—	—	-404.9
<u>Boxes 39, 40, and 41</u>								
M0094	36732.4	35583.3	—	—	40.5	0.9	1107.7	—
M0095	32858.0	32422.4	—	—	83.0	9.3	343.3	—
Enrichment 1	69590.4	68005.7	—	—	123.5	10.2	—	1451.0
M0096	31239.7	31402.0	—	—	—	5.0	-167.3	—
M0097-8	45154.4	44519.7	—	—	74.2	67.4	493.1	—
Enrichment 2	76394.1	75921.7	—	—	74.2	72.4	—	325.8
ISE Campaign 1	—	—	—	—	—	—	—	1776.8
<u>Boxes 39, 40, and 41 (corrected for redundant entry)</u>								
M0094	36732.4	35583.3	—	—	40.5	0.9	1107.7	—
M0095	32858.0	32422.4	—	—	83.0	9.3	343.3	—
Enrichment 1	69590.4	68005.7	—	—	123.5	10.2	—	1451.0
M0096	31239.7	30627.1	—	—	—	5.0	607.6	—
M0097-8	45154.4	44519.7	—	—	74.2	67.4	493.1	—
Enrichment 2	76394.1	75146.8	—	—	74.2	72.4	—	1100.7
ISE Campaign 1	—	—	—	—	—	—	—	2551.7

TABLE IV-A (Continued)

<u>Box 42</u>								
<u>Material Identity</u>	<u>In</u>	<u>Out</u> ^(a)	<u>Sample</u> ^(b)	<u>Clean Scrap</u>	<u>Dirty Scrap</u>	<u>Waste</u>	<u>Oxide Balance</u>	
							<u>Blend</u>	<u>Enrichment</u>
M0094	35139.6	34570.6	190.1	236.4	—	—	142.5	—
M0095	32177.8	31735.2	200.1	254.3	—	38.3	— 50.1	—
Enrichment 1	67317.4	66305.8	390.2	490.7	—	38.3	—	92.4
M0096	31402.0	30221.4	138.5	304.2	—	—	737.9	—
M0097-8	36613.8	35886.3	277.5	258.7	202.0	17.4	— 28.1	—
Enrichment 2	68015.8	66107.7	416.0	562.9	202.0	17.4	—	709.8
ISE Campaign 1	—	—	—	—	—	—	—	802.2
<u>Box 42 (corrected for redundant entry)</u>								
M0094	35139.6	34570.6	190.1	236.4	—	—	142.5	—
M0095	32177.8	31735.2	200.1	254.3	—	38.3	— 50.1	—
Enrichment 1	67317.4	66305.8	390.2	490.7	—	38.3	—	92.4
M0096	30627.1	30221.4	138.5	304.2	—	—	— 37.0	—
M0097-8	36613.8	35886.3	277.5	258.7	202.0	17.4	— 28.1	—
Enrichment 2	67240.9	66107.7	416.0	562.9	202.0	17.4	—	—65.1
ISE Campaign 1	—	—	—	—	—	—	—	27.3
<u>Box 43</u>								
M0094	39600.2	35901.0	32.7	3818.4	—	—	—151.9	—
M0095	31143.4	29243.4	32.0	1493.3	283.1	24.7	66.9	—
Enrichment 1	70743.6	65144.4	64.7	5311.7	283.1	24.7	—	—85.0
M0096	30221.4	26232.5	35.8	3963.5	29.7	0.2	— 40.3	—
M0097-8	35886.3	35016.5	38.0	687.7	—	3.9	140.2	—
Enrichment 2	66107.7	61249.0	73.8	4651.2	29.7	4.1	—	99.9
ISE Campaign 1	—	—	—	—	—	—	—	14.9

(a) to other boxes in process line

(b) out of process line

Table IV-B: Summary of Weight Balances by Box and Master Blend

<u>Individual Values</u>					
<u>Blends</u>	<u>37</u>	<u>38</u>	<u>Boxes</u> <u>39-41</u>	<u>42</u>	<u>43</u>
94	190.2	-366.0	1107.7	142.5	-151.9
95	-208.0	-50.5	343.3	-50.1	66.9
Enr. 1	-17.8	-416.5	1451.0	92.4	-85.0
96	93.3	52.8	607.6	-37.0	-40.3
97/8	-128.0	-41.2	493.1	-28.1	140.2
Enr. 2	-34.7	11.6	1100.7	-65.1	99.9
Campaign	-52.5	-404.9	2551.7	27.3	14.9

<u>Cumulative</u>					
<u>Blends</u>	<u>37</u>	<u>38</u>	<u>Boxes</u> <u>39-41</u>	<u>42</u>	<u>43</u>
94	190.2	-175.8	931.9	1074.4	922.5
95	-208.0	-258.5	82.8	32.7	99.6
Enr. 1	-17.8	-434.3	1016.7	1109.1	1024.1
96	93.3	146.1	753.7	716.7	676.4
97/8	-128.0	-169.2	323.9	295.8	436.0
Enr. 2	-34.7	-23.1	1077.6	1012.5	1112.4
Campaign	-52.5	-457.4	2094.3	2121.6	2136.5

B. Campaign 1 Plutonium Balance

1. Discussion on balance inputs

a. Feed

The makeup of the feed material used in Campaign 1 was rather complex. The plutonium dioxide used in this first campaign was obtained in 1969. Six cans of PuO_2 were used; namely, cans designated as A, B, C, E, F and G. Cans E, F and G were unopened, full cans. On the other hand some of the contents of cans A, B and C had been used in 1969 fabrication activity. It was assumed that the atmosphere in the 3 unopened cans remained the same as when sealed by the shipper. According to information from the shipper (ARHCO) the relative humidity (RH) of the loading box atmosphere was about 4 percent. Another assumption was that the 3 opened cans (A, B and C) had picked up moisture during their use in 1969 fabrication activity, since the RH of the GE plutonium fabrication laboratory is in the 40-60 percent range. Equilibration tests on plutonium dioxide powder, confirmed this, a sharp rise in the powder weight being observed within the first few hours when "very dry" powder was exposed to

a 50% RH atmosphere. It was assumed that the moisture content for the unopened cans was different than that of the opened cans of Campaign 1 feed material. This implies that the plutonium concentration, defined as gPu/g material, would be different for feed material from cans A, B and C than for material from cans E, F and G. From equilibration experiments the change in the plutonium concentration in PuO₂ can vary between 0 and 1 percent depending upon temperature, humidity, particle size and surface area. For Campaign 1, samples were taken for wet chemistry analysis of the plutonium concentration from cans B, C, E and G. It was generally agreed by ISE-TR and GE personnel that any PuO₂ samples sent to the plutonium analytical laboratory would reach the equilibration plateau before they were analyzed and therefore any "as received" plutonium concentration values were assumed to be at equilibrium with respect to the analyzing environment. Before the samples were destructively analyzed for plutonium content the samples were "dried" at a selected temperature to drive off moisture. Plutonium concentration values obtained under "dried"

conditions were supposed to represent the dry received plutonium dioxide. It was interesting to note that the average value of the 3 sub-samples taken from a composite sample (which was made from equal portions of samples from cans B, C, E, and G) was 87.27% Pu for a "dried" condition where the drying temperature was in the 200-300 °C range. The average value for these same 3 sub-samples was 88.09% when subjected to a temperature in the 700-800 °C range.

It is clear that the definition of the word "dried" may vary depending on conditions, and that selecting a drying temperature that will condition the sample such that it may be considered representative of the feed must be carefully done and strictly adhered to.

After the four cans were sampled, two of the cans (B and E) were opened to the box atmosphere and allowed to absorb moisture. Can B percent weight gain during this equilibration test was slightly lower than that observed from the contents of can E. This result was expected because can B had been previously opened and exposed to higher RH atmosphere.

In contrast to this, it was surprising to note that two containers of material which made up Can E contents did not have similar percent weight gain. Container E1 had a weight gain of 0.77% and container E2 a value of 0.88% after the material was equilibrated. It was noted that the sample taken to represent can E came from container E1. From the information obtained from the weight gain (equilibration) test it appeared that the PuO_2 powder in container E1 has about the same moisture content as did the powder in can B. No information was available to explain this condition.

The following assumptions were made prior to wet chemistry analysis on Campaign 1 feed material:

Can C: The values for the special third laboratory samples (in this case NBL) and the remaining PuO_2 , after completion of Campaign 1, would contain more moisture than the process feed and the Plutonium Analytical Laboratory sample values.

Can B: The gPuO_2 value of 330.9, which was the amount in can B that went into the

second enrichment, should contain more moisture than when the can was introduced into the fabrication line.

The largest obstacle in determining a meaningful feed value and its LE for Campaign 1 was the selection of the proper % Pu value for the feed which consists of:

- partially equilibrated material (cans A, B, C),
- partially-plus equilibrated material (cans B & C outgoing), and
- as-received-from-shipper material (cans E, F, G).

Because of the above it was not possible to compute a meaningful gPu in the feed or its associated limit of error by using the wet chemistry analysis data. The data from calorimetry NDT was used to make such determinations. The details of calorimetry NDT are described above in Section II.

b. Product

The grams of plutonium in the product and its uncertainty were computed using the wet chemistry analyses. There were two master blends of material made up for each of the two enrichments. The two master blends within a given enrichment were supposed to be the same both as to concentration of plutonium and as to the sampling error in Pu concentration measurements. For the first enrichment, the Pu concentration of the master blends M0094 and M0095 were the same according to a test but the sampling errors were different according to an F test* on the observed variances of the measurements of the product plutonium concentration. The Pu concentrations and sampling errors of the master blends of the second enrichment, M0096 and M0098, were not statistically different. Therefore, the grams of M0094 mixed oxide and the M0094 plutonium concentration data were kept separate from the M0095 information. For the second enrichment all the information was pooled to-

*The F statistic is the ratio of the variances of two samples. The F test provides the probability that the two samples came from normal distributions with the same variance.

gether; i.e., there was an average value for each of the two master blends. It was important that the data be treated properly not only to be able to determine total grams plutonium in the product but also to insure that the proper uncertainty would be obtained on the product. The average Pu concentration of M0094 was multiplied by the M0094 grams of mixed oxide that were put into the rods to obtain the M0094 total grams of plutonium. The variance of the total grams is the sum of the variances of the individual weight measurements. The total weight uncertainty was then converted to a percentage and linearly added to the average Pu concentration percent value to get the total percent uncertainty on the M0094 grams plutonium in the product. The same procedure was followed for M0095. To obtain the uncertainty on the total grams plutonium in the first enrichment, the uncertainty values, in absolute units, were propagated in the normal manner. Normal error propagation techniques were again used to combine the two enrichment uncertainties to obtain the Campaign 1 total product uncertainty. As one would expect, the normal propagation of error

technique was applied in all situations except where the total grams of mixed oxide is multiplied by the average Pu concentration value. The treatment of errors in this case requires a systematic procedure because a collection of values is being multiplied by a constant.

It was planned to determine the grams Pu and its uncertainty from data obtained by gamma scanning fuel rods to compare with the wet chemistry data. Unfortunately the analysis of the gamma scanning data was not completed in time to be used in this report.

c. Measured Discards (Waste) and Scrap

The waste and scrap materials resulting from the fabrication of mixed oxide pellets for Campaign 1 were measured for plutonium content by neutron coincidence counting. The standards used for calibration purposes were fabricated from existing mixed oxide materials. The Pu content reported from a previously made wet chemistry analysis on the oxide was used for the standard. The LE on the gPu in the standards ranged from 0.4% to 1.9% for scrap standards and for waste standard was 3.2%

or 3.5%. Most of the LE is due to the uncertainty associated with the plutonium concentration determination.

The coincidence method observes neutrons from the spontaneous fission of Pu238, Pu240, and Pu242. Other isotopes, such as U238, have spontaneous fission but they do not make a significant contribution. The calibration curves for the neutron coincidence counting of waste and scrap were derived from the following data: ordinate is coincidence counts from standards corrected for accidental counts, dead time, and background counts; abscissa is the g Pu240 equivalent in the standards. All data were weighted according to counting statistics. In determining the gPu240 equivalent the spontaneous fission ratios were obtained from GE calculations on data in Plutonium Handbook, Vol. 2, by Wick.

It was found that the neutron equipment was not stable over the three month period that the Campaign 1 waste and scrap material was counted and therefore it was not possible to use just one or two calibration

curves. A study of the scrap standards data showed that data taken on the following dates could be pooled to form calibration curves for the four periods:

- (1) October 7-13;
- (2) November 10-13;
- (3) December 1-16;
- (4) December 23-28.

Some pooling of scrap standards data was necessary in any event because on most days that Campaign 1 material was counted the standards measured did not cover the counting range of the samples. Even with the pooling the calibration curves did not cover some of the "hotter" scrap packages. A weighted regression analysis (linear least squares fit to $y = ax + b$) was performed on scrap standards data for each of the four periods. Coincidence counting statistics were used as weights.

Data analysis on the very weak waste standards did not show any statistically significant trend. Therefore all 64 neutron coincidence measurements on waste standards PIP 23-28 were used in one weighted regression.

d. PuO₂ Feed in Ending Inventory

Can C contained 135.7 gPuO₂ at the end of the campaign. The sample value obtained at the beginning of the campaign was used in computing the gPu value. Since only one determination was made on can C, a LE could not be calculated. In lieu of this, an estimated value of 1% was used.

e. Samples

The plutonium dioxide samples, and the green and sintered mixed oxide samples removed from the fabricating line, were accounted for in terms of gPu by using the pertinent analytical values to compute the gPu as well as the LE.

2. Basic Data and Formulas

a. Feed - Calorimetry

Table IV-C shows the isotopic abundances and watts/gram values that were used to compute the gPu values shown in Table IV-D. The wattage measurement from the calorimeter is divided by the total watts per gram to obtain the grams of plutonium. The total watts per gram included the contribution from Am²⁴¹ as well as the plutonium. The Pu²³⁸ and Am²⁴¹ abundances shown in Table IV-C are corrected vendors values.

The standard deviation (σ) of an individual calorimetry measurement is 0.12 gPu (from GEAP-12114-3). The 2σ associated with the total gPu from calorimetry for ISE Campaign 1 material is $.24 (1/6 + 3 + 2(1/2))^{1/2} = .5$ gPu.* To obtain the total LE one must determine the systematic error associated with the isotopic content. For Campaign 1 material the main plutonium isotopes which contribute to the heat are Pu238, Pu239, Pu240 and Am241. To determine this systematic error one observes the percent change in a given gPu value when the isotopic abundances are varied within their uncertainty range. The uncertainty on the Pu238 and Am241 abundances which contribute 17 and 13 percent of the heat, respectively, resulted in a 4.0% LE on the gPu in the feed material.

b. Product - Wet Chemistry (Coulometry)

Table IV-E shows the average Pu concentration and its uncertainty for master blends M0094 and M0095 and for enrichment 2.

*Six measurements on one container, one measurement on each of three containers and two measurements on each of two containers.

Table IV-F gives the total grams of mixed oxide for the blends as well as the LE.

The gPu in the product by enrichment and campaign is shown in Table IV-G.

c. Measured Discards (Waste) and Scrap

The raw data used for calibration purposes and for the Campaign 1 waste and scrap appears in the PIP Third Quarterly Report (GEAP-12114-5).

A study of the neutron coincidence data on scrap standards PIP-8, 9 and 10 indicated a drift problem. Weighted regressions on these standards showed that the slope was increasing as a function of time. The information given in Table IV-H was used as calibration data for the Campaign 1 waste and scrap. It should be pointed out that scrap standards PIP 11, 12, 14 and 15, which were used only on December 16, 23 and 28, appeared to produce a different calibration curve. This phenomenon should be examined more closely in the future since, such a situation, if disregarded, could cause as much as a 5% change in the scrap value.

Additional information on the waste and scrap standards is given in Table IV-I.

Table IV-C: Calorimetry Data

<u>Isotope</u>	<u>Abundance</u> gms/gm	<u>Watts/gram</u>
Pu 238	0.103*	0.566420 ¹
Pu 239	87.15	.001924 ¹
Pu 240	10.09	.007089 ¹
Pu 241	2.38	.003348 ¹
Pu 242	0.28	.0001146 ¹
Am 241	.41*	.11450 ²

¹Mound Laboratory

²Journal of Inorganic and Nuclear Chemistry, Vol. 29, pp. 2659.

*Vendors values corrected to August '70.

Table IV-D: Feed Can gPu Values

<u>Feed Can</u>	<u>gPu</u>
A	485.2
B	586.4
C	817.7*
E	988.8
F	1019.0
G	1016.0*

TOTAL introduced into the fabrication line: 4919 gPu

*Cans C and G were sampled in box 38 before calorimetry. Add 6.3 gPu to total to obtain the total amount into the fabrication.

Table IV-E: Product Average Pu Concentration and LE

<u>Enrichment</u>	<u>n</u>	<u>Average Pu concentration</u>	<u>LE</u>
1 M0094	13	0.03548	0.00020
M0095	12	0.03536	.00035
2 M0096, 98	26	0.03427	.00011

Where n is the number of measurements.

Table IV-F: Product grams of Mixed Oxide

<u>Enrichment</u>	<u>grams of mixed oxide</u>	<u>LE</u>
1 M0094	26323.2	0.8
M0095	28993.3	0.7
2 M0096, 98	61249.0	0.9

Table IV-G: Product gPu and LE

<u>Enrichment</u>	<u>grams Plutonium</u>	<u>LE</u>
1	1959.1	11.5
M0094	933.9	5.3
M0095	1025.2	10.2
2	2099.0	6.8
1+2	4058.1	13.4

Table IV-H: Waste and Scrap Regression #Parameters

<u>Material Designation</u>	<u>Slope</u>	<u>SD Slope*</u>	<u>Intercept</u>	<u>SD Intercept*</u>	<u>Cov**</u>
Waste	14035	402	6.2	5.3	-1991
Scrap**					
Period 1	13950.	71	23	81	-5411
Period 2	14228.	137	-6	156	-20305
Period 3	15152.	120	-126	137	-15627
Period 4	15968.	110	-11	126	-13241

* SD - Standard Deviation

** Cov - Covariance between slope and intercept.

*** Data from scrap standards PIP 8, 9, and 10.

Counts per unit time versus grams Pu240 equipment

Table IV-I: PIP Standards Information

<u>PIP Standards</u>	<u>gPu</u>	<u>Pu240 Eq*</u>	<u>LE on gPu</u>
Scrap 8	1.150	0.126	1.1%
9	2.298	0.251	1.0%
10	11.235	1.229	1.9%
11	2.632	0.288	0.7%
12	5.256	0.575	0.7%
13	24.583	2.689	0.7%
14	4.089	0.447	0.5%
15	8.227	0.900	0.4%
16	40.899	4.474	0.4%
Waste 23	0.0139	0.00145	3.5%
24	0.1352	0.0141	3.2%
25	0.0151	0.00158	3.5%
26	0.1358	0.0142	3.2%
27	0.0141	0.00148	3.5%
28	0.1353	0.0142	3.2%

*Pu240 equivalent = g Pu240 + 3.23 (gPu238) + 1.61 (gPu242). The scrap standards have the same isotopic composition as does the Campaign 1 scrap material. The gPu240 eq./gPu used was 0.1094 for scrap and 0.1046 for waste standards.

The following relationships and values were used to calculate the coincidence counts for both the standards and campaign 1 waste and scrap samples.

y = net corrected coincidence counts

R = Real counts (live time)

A = Accidental counts (live time)

$R+A$ = real plus accidental counts (live time)

G = gross counts (live time)

$D = 8.5 \times 10^{-6}$ sec (dead time)

$S = 129 \times 10^{-6}$ sec (gate time)

$t = 600$ sec

$G_c = G - \frac{D+S}{S} A$ are the gross counts corrected for the accidental counts.

$y = [(R + A) - (A)] / (1 - DG_c/t)^3 - \text{Average Background}$

The dead time correction is very small on the waste standards. The average background value is very small compared to the net counts on the scrap standards.

An average background value of 4 was used because the within day variation was about the same as the day to day variation.

The weighting was according to coincidence statistics.

$$\sigma^2 = (R + A) + A + 12$$

The value 12 represents the variance from the distribution of background values.

In most cases this value was small compared to the sum of the other two values.

The following calculations were used to obtain the gPu240 equivalent and its LE on the ISE Campaign 1 waste and scrap.

$y = aX + b$ general equation From calibration data:

a - slope
b - intercept

$$X_i = (y_i - b)/a$$

σ_a^2 - slope variance

$$X = \sum X_i = (\sum y_i - nb)/a$$

σ_b^2 - intercept variance

cov (a,b) - covariance between slope and intercept

y_i - net corrected coincidence counts of sample i.

X_i - calculated gPu240 eq. of sample i.

$$\frac{\sigma_X^2}{X^2} = \frac{\sigma_{\sum y}^2 (\sum y - nb)}{(\sum y - nb)^2} + \frac{\sigma_a^2}{a^2} - \frac{2 \text{Cov} (\sum y - nb, a)}{(\sum y - nb)(a)}$$

$$= \frac{\sigma_{\sum y}^2 + n^2 \sigma_b^2}{(\sum y - nb)^2} + \frac{\sigma_a^2}{a^2} - \frac{2[\text{cov}(\sum y, a) - n \text{cov} (b, a)]}{(\sum y - nb)(a)}$$

$$\sigma_X^2 = \left(\frac{\Sigma y - nb}{a} \right)^2 \left[\frac{\sigma_{\Sigma y}^2 + n^2 \sigma_b^2}{(\Sigma y - nb)^2} + \frac{\sigma_a^2}{a^2} + \frac{2n \text{ cov } (a,b)}{(\Sigma y - nb)(a)} \right]$$

For the waste there was only one period but there were four periods for the scrap. To obtain the total gPu240 equivalent for the scrap and its variance the following procedures were followed:

$$X_T = \sum_j^4 X_j$$

$$\sigma_{X_T}^2 = \sum_j^4 \sigma_{X_j}^2$$

The gPu240 equivalent results for Campaign 1 waste and scrap are given in Table IV-J. To obtain the gPu value, the gPu240 eq. value was divided by the gPu240 eq./gPu ratio.

d. PuO₂ Feed in Ending Inventory

The sample taken from Can C at the beginning of the campaign gave a value of 86.4% Pu. A limit of error value was estimated to be 1% for the concentration.

Table IV-J - ISE Campaign 1 Waste and Scrap gPu240
Equivalent Results

Waste

<u>Number of Samples</u>	<u>gPu240 eq.</u>	<u>σ gPu240 eq.</u>
72	2.961	0.063

Scrap

<u>Period</u>	<u>Number of Samples</u>	<u>gPu240 eq.</u>	<u>σ^2 gPu240 eq.</u>
1	15	9.566	.00411
2	1	7.130	.00934
3	17	5.900	.01289
4	18	37.188	.02991
TOTAL		59.784	.05625

The weight of PuO₂ in Can C was 135.7 g with a LE (weight) of 0.1%.

Results: 117.2 ± 1.2 gPu.

The 11.1 gPuO₂ sample taken from Can C should be treated with the same Pu concentration value and uncertainty.

e. Samples

The following table summarizes the average plutonium concentration and its LE for both the plutonium dioxide and mixed oxide samples.

<u>Material</u>	<u>Number of Samples</u>	<u>Average Pu conc.</u>	<u>LE</u>
Mixed Oxide:			
Green (M0094, 95)	6	0.0347	.00075
Green (M0096, 98)	6	0.0339	.00033
Sintered (M0094)	13	0.0355	.00020
Sintered (M0095)	12	0.0354	.00035
Sintered (M0096, 98)	26	0.0343	.00011
Plutonium dioxide:			
Analytical samples	7	0.853	.0060

3. Values and Associated LE

The following table, IV-K, lists the gPu values and the associated limit of error values for all of the inputs to the Campaign 1 measured material balance. It should be noted that the measured discards value (waste) does not include the absolute filters. The LE value associated with the measured discards includes a 3.2% uncertainty in the waste standards. LE associated with the scrap in ending inventory includes a 1% uncertainty associated with the scrap standards Pu value.

4. MUF and LEMUF

The MUF is 112 gPu with a limit of error value of 198 gPu. As one can observe from table IV-K the LEMUF is due almost entirely to the LE FEED which in turn is due to the uncertainty associated with the isotopic abundances. This LE (4.0%) was considered unreasonably high. Discussions with NBL personnel on this subject revealed that NBL capability for determining uncertainties in similar cases resulted in a LE for the feed of 0.6%. Thus if NBL uncertainty values were applied to Campaign 1 feed, the LE value associated with the feed would be 31 gPu instead of 197 gPu. The LEMUF value would be 36 gPu instead of 198 gPu.

Table IV-K: gPu Material Balance and LE

<u>Balance Input</u>	<u>gPu Value</u>	<u>LE Value</u>
Feed (Calorimetry)	4919	197
Product (Coulometry)	4058	13
Measured Discards	27	2
Ending Inventory		
Scrap	546	10
PuO ₂ Feed	117	1
Samples		
Mixed Oxide	34	0
Plutonium Dioxide	25	0
MUF	112	198

V. Summary of Findings

A. Mixed Oxide Weight Balances

This type of box balance is useful for locating where major losses occur within a processing area. On a master blend basis, where partial cleanout occurs between blends, unique material identity is necessary in order for weight balances to be useful.

B. Campaign 1 Plutonium Balance

1. The MUF is 2.4% of throughput. This value appears to be high compared to the GE-VNC annual survey values by a factor of two.
2. Isotopic abundance determinations will have to be improved in order for calorimetry to be a useful nondestructive technique in material accounting.
3. Procedures need to be developed for sampling feed materials where moisture effects are present.
4. Procedures are needed for all NDT methods involving standards. This includes the making of standards, testing equipment, calibration practices, etc.
5. The LE of the scrap value may conceal a bias when only a limited number of standards are used in preparation of neutron coincidence calibration curves.

6. Neutron coincidence counting of dirty scrap and waste permitted GE-VNC to obtain a measured material balance.
7. No comparison between non-destructive and destructive techniques could be made during ISE Campaign 1.
8. NDT covariance effects. In cases where the same calibration curve is used in determining the gPu for many samples the resulting standard deviation on the total will be a factor of two or three smaller if the covariance effect* is neglected.

C. Campaign 1 Impact on Conduct of Campaign 2

1. Procedures were developed that resulted in meaningful sampling of PuO₂ feed material. This procedure included the obtaining of representative samples as well as data for calculating the Pu concentration limit of error.
2. The effects of biases among the various measurement methods on cross-comparative analysis were recognized. As a result, a replicate sample - replicate analysis plan was prepared and in-

* Situations may exist where certain values and their associated errors are related. These situations are called dependent. Covariance is a mathematical expression which quantifies the degree of dependence of the errors.

corporated in Campaign 2 for identifying and controlling measurement bias in gamma counting, neutron and coulometry assay.

D. Importance and Role of NDT

1. The neutron coincidence counting method coupled to good sound procedures should allow all plants handling plutonium to obtain material balances based completely on measurements.
2. Detailed standard procedures should be part of each NDT package. This should include details for proper data analysis.

E. General

1. Educational programs are needed so that plant personnel responsible for material balance accounting can expand their data analysis capability and handle error propagation in a correct manner. This data analysis item particularly applies to the non-destructive testing field. For example at GE-VNC the neutron coincidence counting equipment drifted during the time ISE Campaign I scrap was measured. If this fact was ignored a bias of about 30 gPu would be reflected in the ISE Campaign 1 measured material balance.
2. The GE-VNC personnel responsible for calculating material balances were not experienced at data

analysis and statistical treatment of measurement data, particularly that taken from NDT measurements. If these people are typical of the industry it is possible that an extensive education and training program will be a necessity in order to have licensees calculate meaningful material balances.

3. The Integrated Safeguards Experiment was designed to test the usefulness, to safeguards, of material balance accounting techniques which employ data based completely on measurements. The plan was to utilize non-destructive testing techniques to accomplish this result and it was shown that a completely measured material balance was feasible. Feasibility, however, does not mean that it is practicable for routine safeguards use. To be useful the utilization of NDT should (1) improve the accuracy of the material balance, (MB) (2) greatly improve the timeliness of the MB, and (3) provide a cost incentive to the user. To determine whether or not the use of NDT improved the accuracy of the MB it was necessary to compare results of Campaign 1 to those of a similar and previous GE Campaign which did not utilize NDT. Since historical data made

available by GE for this purpose were not dependable, it was not possible to make a comparison.

4. The inexperience of the GE plant people in both the operation of NDT equipment and in analyzing the data taken coupled with the lack of knowledge in preparing standards resulted in poor measurements and much delay in striking material balances.
5. In-so-far as evaluating the utilization of NDT from a cost incentive view point, there was not sufficient time and funds available to conduct such study during the term of Campaign 1.
6. Also before such study could be made certain economic policy problems must be resolved.

Examples of these are as follows:

- (a) Is it necessary to measure all material streams simply to satisfy the requirements of a completely measured MB?

Obviously if the operation is a small one (such as the one conducted by GE) and the Pu content of wastes and discards is extremely small, very little is gained by the measurement.

- (b) At what points in a production process should measurement be made? This pro-

blem would not be a point of contention if Task E (determination of process loss by unit operation) could have been accomplished. The reasons why this objective was not accomplished are explained in this report and the deficiencies in the measuring and sampling procedures which caused the cancellation of this task are now well known and can be used to good advantage by the nuclear industry.

7. Conclusions drawn in this report to effect means of improving material control to satisfy the requirements of safeguards could only have been made by carefully studying, in detail, the process operation at GE. It is believed that these conclusions may be applied generally throughout the industry and from this point of view, Campaign 1 has been very successful.
8. The MUF reported for this campaign is 2.4% of material throughput. This is much too high for practical operation. This campaign has shown however, that much of this MUF can be attributed to deficiencies in measurement and sampling methods. Other experiments, with improved NDT instrumentation, conducted by

TSO/Brookhaven at an industrial facility similar to the GE operation and TSO studies made of a hypothetical plant operating, under conditions of improved methods of sampling, measuring, record keeping and reporting and data analysis, permits a logical extrapolation of improved performance to the GE plant. Under these conditions the indications point to the possibility of routine material balance with MUF values of about 0.5% of plutonium throughput.

VI. Conclusions

A meaningful measured material balance can be accomplished if the following additional procedures are used:

Introduce NDT measurements for disposable waste and scrap, and for checking feed and products.

Develop standard procedures to be followed by the total industry in making measurements to establish SNM value.

Provide direction for data analysis and calculating LE's.

Possibly stipulate the minimum reporting requirements for maintaining material control.

Establish standard procedures for calibrating measuring equipment.

VII. Acknowledgements

As stated in the introduction of this report, Campaign 1 of the Integrated Safeguards Experiment was conducted jointly by GE-VNC, Technical Support Organization of the Brookhaven National Laboratory, and the Technical Analysis Division, National Bureau of Standards. It is logical then, that a report covering the details of this Campaign should also be the product of the joint effort of the participating members. Such was the case. The task of organizing, drafting and editing the report was accomplished by TAD/NBS. Raw data, furnished as accumulated by GE, was analyzed by TAD/NBS with TSO Assistance. Experimental and fabrication plans, as noted in Section I-E were developed by GE. Technical discussions, covered by Section's II-F and III, were contributed by TSO as well as the calculations reported under Section IV-A. The conclusions drawn in this report reflect the considered opinions of both TAD/NBS and TSO/Brookhaven.

Appendix

Description of Tasks 2A-2G

The total ISE includes complete material balances for independent fabrication campaigns utilizing measurement data for all inputs and outputs with maximum utilization of non-destructive test equipment as demonstrated in the Plant Instrumentation Program. As each campaign progresses to another, refinements to procedures followed in the previous campaign would be added and results of all campaigns compared to note improvement from one to the next.

Since each campaign covered a variety of experiments the conduct to be followed was defined by a series of tasks. Campaign 1 tasks proposed by GE and approved by the ISE Team were as follows:

1. Task 2A

- a. Experimental plan. The campaign was started on August 3, 1970 with the fabrication of 136 kg of mixed oxide fuel containing 90 percent fissile plutonium. Normal production and available PIP-NDT instrument measurements were used to establish a material balance. Standard cleanout procedures were made between enrichments. The following plan was approved for this task:

(1) Box Cleanout

- (a) All nuclear material remaining in equip-

ment and other "clean" nuclear material (recycle and/or scrap) will be composited (for each glove box), weighed, identified and isolated in a container(s). After bag-out, each container will be measured with PIP-NDT gamma or neutron instruments.

- (b) All floor sweepings and other "dirty" nuclear material (scrap) will be composited by glove box (primarily by means of a vacuum cleaner). The nuclear material will be isolated in a container, weighed and identified. After bag-out from the glove box, the container will be measured by PIP-NDT gamma or neutron instruments.
- (c) The interior of each box will be vacuumed and all working surfaces will be wiped down. Used vacuum cleaner bags will be emptied and nuclear material composited with other "scrap." Vacuum cleaner bags will be weighed before and after use. The empty bags will be composited with other "discard" material (for each glove box). All "discard" material will be bagged-out of the glove box and measured with PIP-NDT gamma or neutron instruments, if feasible.

(d) Exhaust filters will be replaced with new weighed filters before and after each campaign. Filters will be weighed after each campaign to permit estimation of nuclear material content. After bag-out from the glove box, the filters (discard) will be measured with PIP-NDT gamma or neutron instruments, if feasible.

(2) Feed

A record will be maintained of all nuclear materials entering the glove box line. All nuclear material entering the system will have the following data available, if applicable: net weight to 0.05 grams, SS weight, fissile weight, Pu/PuO₂ analysis, U/UO₂ analysis, Pu/PU+U analysis (production samples), O/M and/or volatiles on feed batches, and impurity analysis or vendor data on feed batches.

(3) Product

A record will be maintained of all fuel materials leaving the glove box line. Sintered fuel certification data will be used to account for all fuel and will include: net weight to 0.05 grams, SS weight, fissile weight, Pu/Pu+U analysis, O/M, and impurity analysis.

(4) Scrap, Recycle and Discards

Nuclear material content for all scrap, recycle and discards will be determined as indicated in Section (1) above.

(5) Data Records

The following information to be made available to ISE Technical Representatives:

- (a) Measured material balance by enrichment based on data records will be maintained to provide material balance calculations from a single data package for each enrichment. This may involve special forms or copies of production records so the data package will be completely documented and "free standing."
- (b) No special samples would be withdrawn. Discard, recycle and scrap material will be measured by single non-destructive test readings.
- (c) Raw data (weights) for nuclear materials transferred into and out of the glove box line will be provided. Complete data for internal (box) transfers within the line will not be available.
- (d) Weekly computer reports on a box basis will be provided when the paper tape inter-

face is completely programmed; limit of error calculations will not be programmed for Campaign 1.

b. Fabrication Laboratory Plan

(1) Box Clean up - Initial

Prior to start-up of Campaign 1 the following procedure was followed:

- (a) Clean out boxes per GE normal procedure.
- (b) Install new preweighed filters, both absolute and pre-filters. Record the weights and dates installed on the established form. Identify each filter plenum with a letter designation; A for the plenum nearest the box window, B for the rear plenum.
- (c) Install new preweighed bags in all vacuum cleaners. Record these weights on the established form.

(2) Standardization - Plutonium Laboratory

- (a) Calibrate all balances.
- (b) Prior to the shift startup, standard weights (available at each weighing station) will be used daily to check each balance which could be involved in SNM (Special Nuclear Material) transfers. Plot the observed value of the standard

weight on the established control chart. After ten values have been plotted, the limits of control will be provided by the Process Control Engineer.

- (c) Run a standard pellet (samples obtained from one special sintering) with each oxygen-to-metal ratio run. Plot the observed value of the standard weight on the established control chart. If the value falls outside of the control limits, rerun the O/M analysis.

(3) Standardization - Analytical

- (a) One standard shall be run each day analyses are performed.
- (b) Maintain a record of each standard run in a notebook.
- (c) If a standard falls outside of the control limits, rerun the analysis and/or re-standardize the equipment.

(4) Material Balance Requirements

- (a) All discards shall be numbered and logged into waste barrels by box number; e.g., designation for Box 37 waste will be 37-1, 37-2, ..., 37-n. Waste packages must be double bagged with a maximum of one gallon or three quart ice cream cartons.

The quart cartons must be arranged in a triangular fashion. At a later date, count all discards with NDT (neutron) equipment.

- (b) At the completion of a campaign, the scrap in each box shall be composited into two groups, "clean" and "dirty" (see Table I-A).
- (c) At the completion of a campaign, composite all recycle material by box.

(5) Blending - Box 37

- (a) Perform NDT measurements (calorimetry) on all plutonia feed prior to introduction into Box 37.
- (b) All SNM introduced into the glove box shall be weighed to an accuracy of 0.5 grams.
- (c) Weigh plutonia (PuO_2) and urania (UO_2) powders before loading into the blender and record the weighing data on the batch makeup sheet.
- (d) The Hammermill heel obtained during a cleanout between an enrichment change is to be weighed, identified by enrichment, and held as scrap in an identified container. Record the weight of the heel on the established form during clean up.

TABLE I-A
MATERIAL CLASSIFICATION

<u>Classification</u>	<u>Measurements</u>	<u>Description</u>
Feed	Chem, Weight, NDT	Plutonia
Product	Chem, Weight, NDT O/M	Fuel Rods
"Dirty" Scrap ¹	Weight, NDT	Floor Sweeps Vacuum Cleaner Bag Residue
"Clean" Scrap ¹	Chem, Weight, NDT	Grinder Mill Heel Granulator Heel O/M Samples Sinter Pellets not Loaded Samples (unused from PAL)
Recycle ²	Chem, Weight, NDT	Green Powder Granulator Heel (if necessary)
Discard ³	Weight, NDT	Prefilters Absolute Filters Vacuum Cleaner Bags Sample (used for analysis) Swabs

¹ Scrap - Material which will be stored for reclamation by chemical dissolution.

² Recycle - Material which can be reused in essentially the same form in additional processing steps.

³ Discard - Material destined for burial.

- (e) Record the net weight of the master blend on the batch makeup sheet after reblending and weighing.
- (f) Sample the master blend according to the work guide and record the weight of the samples on the batch makeup sheet.

(6) Slugging and Granulation - Box 38

- (a) After slugging and granulating the powder as determined by a sintering test, clean the granulator. The heel shall be sealed in a can, identified by enrichment, weighed and held as scrap. Record the weight of this material on the form during cleanout. This material can be hand screened if required for pellet fabrication.
- (b) Handle all scrap, recycle, and discards per item (4) above.

(7) Pellet Pressing - Box 39

- (a) Press the heel in the feed shoe by hand after the completion of fabrication of a master blend.
- (b) Check the tare weights of each sintering boat at the start of each enrichment. Record this data in the Box 39 log book.
- (c) Weigh the loaded boats prior to sintering.

Record the weight on the furnace log and the green sintering data sheet.

- (d) All scrap, recycle, and discards shall be handled as specified in item (4) above.

(8) Sintering - Box 40

No data required.

(9) Grinding - Box 41

- (a) Record the weight of the pellets before and after grinding and the grinding loss by sintering boat (i.e., firing number) on the sintering data sheet.
- (b) The grinding residue obtained from the large vacuum cleaner shall be collected and identified by weight and enrichment and sealed in cans. This weight shall be recorded on the established form during cleanouts.
- (c) Handle all scrap, recycle, and discards per item (4) above.

(10) Inspection - Box 42

- (a) After gaging, the specification pellets shall be accumulated in sealed cans and identified by batch, weight and firing number.
- (b) Record the weight of the pellets taken for samples on the established form.

- (c) Make calorimetry and neutron measurements on every second Pu/Pu+U sample. These samples must be individually double bagged.

(11) Loading - Box 43

- (a) Record the fuel rod weight by batch number of the travel cards.
- (b) Handle scrap, recycle, and discards per item (4) above.
- (c) Make NDT (gamma) measurements on all rods per the PIP schedule.

(12) Box Clean Up - Between Enrichment

- (a) Clean boxes per GE normal procedures.
- (b) Make NDT (neutron and calorimetry) measurements per PIP schedule on scrap (excludes samples) and recycle.

(13) Box Clean Up - Between Campaigns

- (a) Clean boxes per GE normal procedures.
- (b) Remove all filters, both absolute and pre-filters. Record the filter weights and the dates removed on the established form.
- (c) Install new preweighed filters, both absolute and prefilters. Record the filter weight and dates installed in the box log book.
- (d) Make NDT measurements (neutron and calorimetry) on all scrap (excludes

samples) and recycle per PIP schedule.

2. Task 2B - Data Handling Methods

Systems of measurements and records keeping will be developed and tested for implementation during fabrication campaigns. Methods will be developed, programmed and tested for editing, storage and retrieval of data for (a) inventory, (b) calculation of systematic and random errors for measured quantities, and (c) determination of limits of error of all measurements.

3. Task 2C - Data Acquisition System

The Plutonium Fuels Laboratory is developing a data acquisition system to maintain material balances and inventory control throughout the complete fabrication cycle. This system consists of a Raytheon 703 computer with 8000 words of core memory and three teletypes for direct data input from various locations within the Plutonium Laboratory. It is hoped that this system will ultimately establish a material balance system in which the limit of error is defined by the measurement capability.

4. Task 2D - Program Review and Progress Planning

This Task consists of an orderly reappraisal of results and a replanning of the next campaign with the continual support of the sponsor. In this way, new technology may be factored into future campaigns and effort may be diverted from non-productive areas to

more fruitful endeavors. A report will be issued for each program review meeting. Periodic meeting will be held between interested parties and conduct of Campaign 1 will be discussed and decisions made for refinements and changes in future campaigns.

5. Task 2E - Weight Loss by Unit Operation

The objective of Task 2E is to obtain information on process losses and loss functions for several unit operations within the GE-VNC oxide fabrication line. To attain this objective several batches of material would be processed through a given unit operation with a requirement that the operation starts clean and ends clean. For each batch through a given unit operation a gram plutonium balance would be constructed, permitting determination of batch MUF and LEMUF values. To be successful, i.e., for it to demonstrate process loss, the LEMUF value must be small in relation to MUF; otherwise the MUF value would not be significant and process losses would be virtually undetectable.

6. Task 2F - Material Unaccounted For (MUF)

MUF for mixed oxide on a box basis, and MUF and LEMUF on a gram plutonium basis for the entire process line will be calculated for this campaign.

7. Task 2G - Accuracy and Precision Data

This task involves the preparation of a non-proprietary report indicating accuracy and precision for measure-

ments. This will include:

- (a) type of material,
- (b) kind of instrument,
- (c) measurement method,
- (d) calibration method and frequency,
- (e) calibration standards used,
- (f) computational procedures used on measurement and calibration data,
- (g) accuracy (bias) of each method, and
- (h) precision for each method.

