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UNION CARBIDE CORPORATION

P.O. BOX 324, TUXEDO, NEW YORK 10987

TELEPHONE: 914-351-2131

Regulatory

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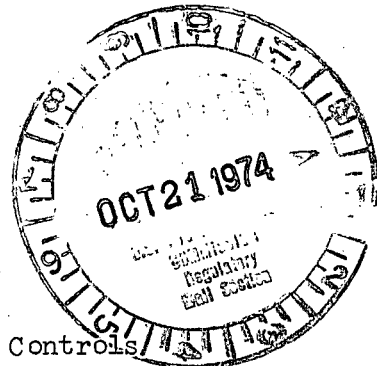
CORPORATE RESEARCH LABORATORY

October 17, 1974

U.S. Atomic Energy Commission
Materials & Plant Protection Branch
Directorate of Licensing
Washington, D.C. 20545

Attention: Mr. C. N. Smith

Dear Sir:

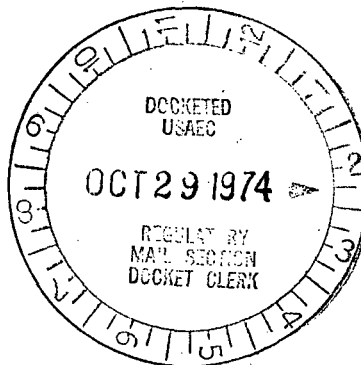


Enclosed please find Section C & F "Measurements, Statistical Controls and Inventory" of the Fundamental Materials Controls and Nuclear Material Safeguards Procedures for our Sterling Forest Laboratory.

These sections have been arranged in accordance with your outline "Guidance in Preparing Material Control and Accounting Plans," and, as requested, are being submitted as a single document. Section 4 of this enclosure is the same as Section B of our partial FMC submittal of 10/4/74. It has been repeated herein for continuity.

As a result of our most recent evaluation of inventory procedures for accuracy, one basic change has been made to our method of assay of irradiation targets. To date these targets have been assayed by weighing. In the future we intend to assay by radiometric analysis. We consider it proper to incorporate this change into the FMC at this time. Therefore, the following changes should be made to the FMC:

- a. Section B., par. 1, line 4. delete: "determined by weighing." add: "measured by gamma-counting"
b. Section E., par. 3, line 2. delete: "weighing" add: "gamma-counting"
c. Section E., par. 4, line 2. delete: "weighing and"



Thank you again for your consideration.

Very truly yours,

UNION CARBIDE CORPORATION
STERLING FOREST LABORATORY

J. J. McGovern

Manager, Radiochemical Production

2122

JJMcG:eb
end

SNM MEASUREMENTS, STATISTICAL CONTROLS AND INVENTORY
(Sections C and F of SFRC FMC)

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APPENDIX C

APPENDIX D

SNM MEASUREMENTS, STATISTICAL CONTROLS & INVENTORY
(Sections C & F of SFRC FMC)

1. Tamper Safing

The current operation at the Union Carbide Sterling Forest Laboratory is such that SNM is not stored for any appreciable time. It is put into process very shortly after it is received. Tamper safing devices are considered unnecessary in the present system.

2. Records Control

2.1 The records which are maintained for the accounting of SNM consist of a Master Log of all SNM possessed under SNM 639 license and individual logs of the SNM in process in each Material Balance Area (MBA).

2.1.1 The Master Log is maintained so that the quantity and distribution of all SNM on Site is recorded and maintained current.

2.1.2 In addition to the Master Log, each MBA maintains a concurrent log of material entering and leaving each MBA.

2.2 Control: Control of internal transfers is exercised by the Site Accountability Officer or his designee. When material is transferred into process or storage it is recorded in the Master Log. Each transfer between MBA's is also recorded in the appropriate MBA logs.

2.3 Signature Requirements: The record of each transfer is signed by the responsible authorized individual in the MBA's to and from which each transfer is made.

2.4 Storage: The Master Log shall be stored in suitable files maintained by the Site Accountability Officer (SAO). The records of each material balance for each inventory will also be stored by the SAO.

2.4.1 Material balance records will be in the form of a separate inventory record in which will be recorded the identity of all items sampled and subsequent calculations showing material on hand at inventory time and LEMUF and MUF determinations. Adjustments to MBA records and the Master Log will be recorded in those logs.

2.4.2 Access to inventory records and the Master Log will be controlled by the SAO. Access to the MBA logs will be controlled by the authorized individuals in each area.

3. Item Control

The SNM in each MBA shall be organized and identified for easy recognition.

- 3.1 a. The material in the UO₂ feed MBA shall be kept in batches, identified and segregated as they are received from the SNM supplier.
- b. The material in the Plating Encapsulation Quality Control MBA will be identified according to a plating solution batch number, a waste solution batch number, or a plated target serial number.
- c. The material in the Reactor MBA will be identified according to a plated target serial number, cross referenced to a Reactor irradiation control number.
- d. The material in the Radiochemical Operations MBA will be identified according to a chemical process batch number, cross referenced to the plated target serial numbers.

3.2 Each inventory will be recorded on a form showing the location and identification of each sample taken. Reports of sample analyses and bulk determinations will also be recorded on the inventory report form. The LEMUF and MUF calculations will also be made on each inventory report form (see Appendix D).

3.3 Transaction Records: The combination of the Master Log Purchase Order file and High Level Radioactive Waste Shipment records will give a history of the source and disposition of all items of SNM.

4. Facility Description

Highly enriched uranium oxide (UO₂), received from an SNM supplier, is dissolved to form a solution. The uranium is then plated out of solution in a layer on the inside of steel tubes. The amount of SNM plated in each tube is measured by γ -counting. The tubes are welded closed. After quality control measurements, the sealed tubes are irradiated in a nuclear reactor. Irradiated tubes are transferred to shielded hot cells where they are opened, the uranium is dissolved, and the desired products are chemically separated. After some months decay, the uranium solution, still highly radioactive, is packaged for shipment to a licensed burial ground.

The flow diagram (Appendix C) shows the sequence of operations, the waste streams, and the locations in the process where SNM logs are maintained (Material Balance Areas (MBA)). The basis for these MBA's are as follows:

- a. UO₂ Feed area is separated by location and administration. Access to this area is controlled by the Health Physics department (SNM Accountability Officer).
- b. The Plating Encapsulation and Quality Control MBA is separated by process requirements. The process being the fabrication of finished irradiation targets.
- c. The Reactor Irradiation MBA is separated by process (or function) location and administration
- d. The Radiochemical Operations MBA is also separated by process location and administration.

5. Physical Inventory

5.1 Description: A physical inventory of SNM shall be conducted on a bi-monthly schedule. Such inventories shall be accomplished by measuring ²³⁵U in materials in process other than that SNM in sealed, doubly-encapsulated irradiation targets and in high level radioactive waste which is located inside shielded hot cells. The inventory of SNM in irradiation targets and high level radioactive waste shall be conducted by locating each target and waste batch and recording the ²³⁵U from the previous radiometric assay.

5.2 Procedure Modification: Inventory procedures will be developed by the Manager Radiochemical Operations, Manager Quality Assurance, and Manager Health Physics and they shall be approved by the Nuclear Safeguards Committee. Each inventory shall be planned and conducted under the direction of the Manager Health Physics.

5.3 Organization: The Manager Health Physics (SAO) will coordinate the work of all those involved in the physical inventory effort.

The Manager Quality Assurance will be responsible for providing assays of all samples submitted.

The Manager Radiochemical Operations shall identify and provide samples of all materials that must be assayed for each inventory period.

The Manager Health Physics and Manager Radiochemical Operations, or their designees, shall complete the final inventory report and reconcile MUF with regard to LEMUF.

5.4 Schedule: The inventory schedule for SNM possessed under SNM 639 shall be as follows:

February 28 (or 29)
April 30
June 30
August 31
October 31
December 31

Each inventory will be conducted within a few days of the above dates. The exact timing for beginning each inventory will be dependent upon the stage of the Plating and QC process and/or the anticipation of waste shipments or SNM receipts.

5.5 Preparation:

5.5.1 A pre-inventory plan will be agreed upon by the SAO, and all Operations and Quality Assurance personnel who will participate in each inventory. This plan will include:

- a. The time of inventory in each MBA
- b. The cut-off point in each MBA
- c. The number of samples to be taken (where necessary) in each MBA
- d. Designated individuals for performing work

A pre-listing of all inventory items will be made on the inventory report form.

5.5.2 Ordinarily, only waste materials will have to be processed in preparation for inventory. All waste materials must be put into solution in preparation for radiometric assay. All other materials should be in the proper physical form for inventory.

5.5.3 The use of inventory tags is considered to be unnecessary due to the limited number of items involved and the normal process identification system in use.

5.6 Cutoff Procedures: Cutoff procedures will be determined by the normal process flow of SNM.

5.6.1 The timing of the commencement of inventory will normally be controlled by the completion of a normal plating-QC cycle in the Plating, Encapsulation Quality Control MBA. When irradiation targets have been plated, sealed, assayed and doubly encapsulated, an inventory of all MBA's can commence. No movement of material between MBA's will be allowed while inventory sampling is being conducted.

The normal labeling of materials in process is sufficient to preclude items from being omitted or double counted.

5.6.2 The Master Log is in the possession of the SAO, all transfers of SNM must be noted in this log. Since no material transfers will be allowed during inventory sampling, there will be no entries made in the Master Log or the MBA logs.

5.7 Traceability: Each sample for measurement will be labeled with an identification number traceable to the source of the sample. The identification number will be recorded on the inventory report form at the time of sample withdrawal. Assay reports on results of analyses will be identified with the original sample identification number.

5.8 Use of Factors: All in-process and inventory measurements will be made on ^{235}U . Conversion factors will not be required for material balance records.

5.9 Prior Measurements: At inventory time, prior measurements will be used for SNM irradiation targets and SNM in high level radioactive waste solutions inside of hot cells. This practice is considered acceptable because:

a. In the case of irradiation targets:

1. Radiometric analysis is performed at the completion of the plating, encapsulation and quality control process steps.
2. Targets are serialized and doubly encapsulated and the SNM cannot be removed without destroying the outer capsule and inner capsule seal.
3. A rough check of the presence of ^{235}U in a target which has been irradiated is available at the completion of the radiochemical process in the hot cells. It is manifested in a measurement of a fission-product isotope (^{99}Mo) yield from each process.

b. In the case of high level waste solutions:

1. all such waste solutions are kept in separate process batch waste containers. Each is identified and can be traced to the target serial number.
2. The radiation level of a batch of waste is several hundred R/hr at one foot. The radiation level in a cell where waste is stored is several thousand R/hr.
3. The only access to this waste would be through a hot cell conveyor system. During off hours, the building is maintained locked with an intrusion alarm on all entrances. The manipulators and conveyor system for the hot cells are kept locked.
4. Any waste material containing SNM would have to be shielded with an equivalent of several inches of Pb. Such shielding casks are not readily available nor easily handled.

5.10 Processing Conditions: As mentioned in Section 516, an inventory will be conducted at the end of a Plating, Encapsulation, Quality Control process batch. The only material in process at time of inventory would be targets in the reactor for irradiation or targets in radiochemical processing. In each such case, prior measurements will be used for inventory.

5.10.1 Prior to inventory, all plating apparatus is drained and rinsed. The hold up in the apparatus will be negligible. All waste material in the Plating, Encapsulation and Quality Control process will be gathered and put into solution prior to measurement. Residual waste after clean-up will be negligible.

5.11 Materials on Inventory

- 5.11.1 A description of the typical materials on inventory is as follows:

(see page 6-a)

<u>MBA</u>	<u>Qty.</u>	<u>Form</u>	<u>Assay Method</u>	<u>Bulk Determinations</u>	<u>Sampling Method</u>
UO ₂ Feed	~ 1 Kg	Solution	Radiometric (compare with std)	Precalibrated volumetric vessels	50λ sample in precalib. micropipet. (duplicate)
Plating, Encapsulation, QC	~ 350 gm	Solution) Sealed Targets)	Radiometric (compare with std)	Precalibrated volumetric vessels (for solution)	Same as above
Reactor Irradiation	~ 300 gm	Sealed Targets	Positive identifi- cation (prior Radio- metric assay)	N.A.	N.A.
Radiochemical Processing	~ 1 Kg	Mixed Fission Product Waste Solution or Solidified in Concrete	Positive identifi- cation (prior Radio- metric assay)	N.A.	N.A.

5.11.2 The uncertainty associated with each of the above measurements will be:

<u>Type of Measurement</u>	<u>Random Error</u>	<u>Systematic Error</u>
1. Radiometric assay	$\pm 5\%$ (includes λ pipette)	~ 0 (NBS std. used as reference each measurement)
2. Volumetric determination of bulk solutions	$\pm 2\%$	$\pm 3\%$

5.12. Verification: The identity of each sample and the location of the origin of each will be verified at inventory in that the drawing of samples by Operations personnel will be witnessed by a representative of the Site Accountability Officer.

5.13 Reconciliation: At the completion of the inventory report (see Appendix D), the SNM recorded in the Master Log (as well as the log for each MBA) will be compared with that found in each inventory. Any difference should be within the calculated LEMUF for that period. The Master Log and MBA, as required, will be reconciled to the physical inventory results.

Any appreciable, unexplained difference will be investigated to determine the cause.

If there appears to be a recurring bias which is attributable to a particular measurement, the measuring system will be re-evaluated for accuracy.

6. Limits of Error

6.1 Materials: Referring to Appendix C, the Plant Process Flow Diagram, the typical quantities and compositions of material in the flow stream are as follows:

- 6.1.1 All special nuclear material is 93% enriched ^{235}U .
- 6.1.2 The UO_2 feed is received in batches of 350 g ^{235}U , up to one batch per week. This material upon receipt is immediately placed in solution as uranyl nitrate, approximately 350 g/l.
- 6.1.3 The feed to the plating MBA is uranyl nitrate solution in batches of 45 g ^{235}U , up to 8 batches per week.
- 6.1.4 The finished items (designated "capsules" in the diagram) each contain about 12 g ^{235}U in the form of UO_2 (93% enriched) at the rate of up to 24-30 items per week.
- 6.1.5 Waste is in the form of uranyl nitrate solution, typically 100 g in 1 liter each 2-month period.

6.2 Measurements: All measurements of the special nuclear material are of the ^{235}U isotope via radiometric methods.

- 6.2.1 Feed material is dissolved completely and made up to an accurate volume in a volumetric flask, typically 1 liter of 350 g/l ^{235}U concentration. A 50 microliter sample is taken, dried on a planchet and the ^{235}U measured by gamma-ray spectrometry (185.5 Kev). This result is used for shipper-receiver differences, as well as for process control.
- 6.2.2 Material is transferred from the feed MBA to the plating MBA by taking a measured volume of feed solution, typically 50-100 ml, in a calibrated pipette. The ^{235}U concentration of this solution is determined by gamma-ray spectrometry (see above).
- 6.2.3 The quantity of ^{235}U in finished items (irradiation targets) for transfer from the plating MBA to subsequent MBA's is determined for each item non-destructively by gamma-ray spectrometry using techniques similar to those outlined in Regulatory Guide 5.38.
- 6.2.4 Waste is determined by radiometric measurement of ^{235}U concentration (as above) and visual volume measurement in calibrated vessel.

6.3 Errors:

6.3.1 For the radiometric measurements (gamma-ray spectrometry) of SNM solutions, the random standard error (including sampling) for ^{235}U determination is 5%. This is based on extensive experience with such techniques. As NBS ^{235}U standards are used for calibration, the systematic error will be close to zero.

6.3.2 For volumetric transfers of feed solution, the random standard error is 1%, the systematic error 0.5%. Pipettes are calibrated by weighing.

6.3.3 For radiometric measurements of the ^{235}U content of individual items, the random standard error is 5%.

6.3.4 Waste solution volume is determined with a random standard error of 2%, and a systematic error of 3%.

6.4 Models: For demonstration of LEMUF capability, a plant process model is assumed in which 4 batches of feed are received, 32 transfers are made from feed to plating, and 120 items (capsules) are made over a 2-month period. Batch quantities are as in Section 6.1, above.

6.4.1) The preceding model results in a LEMUF of 79 g of ^{235}U , calculated as follows:

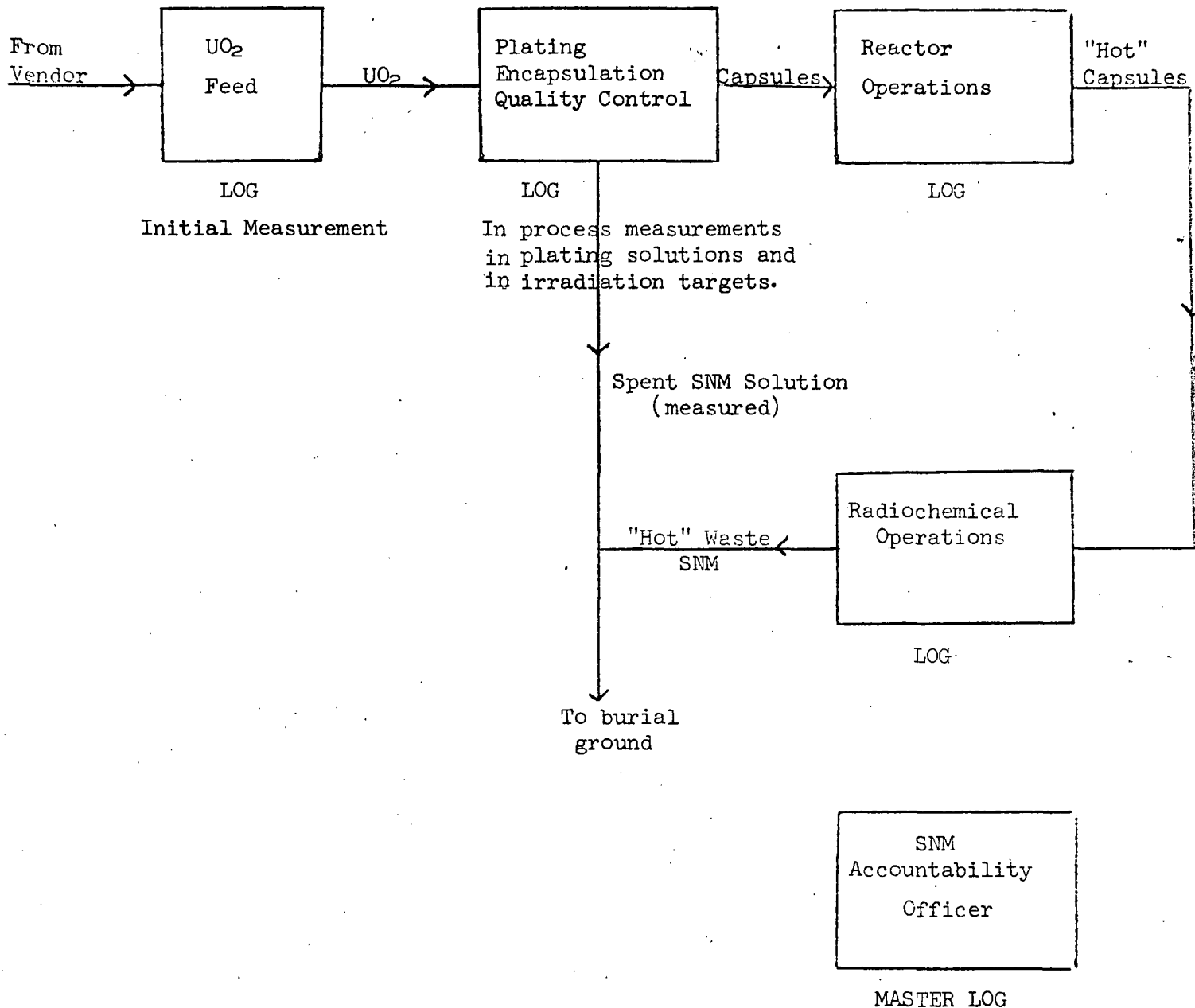
$$\begin{aligned}
 \text{Standard deviation of feed concentration} &= 18 \text{ g/l} \\
 \text{" " " each feed transfer} &= 45 (.05^2 + .015^2) = \\
 & \quad 2.35 \text{ g} \\
 \text{" " " " item content} &= 0.6 \text{ g} \\
 \text{" " " waste content} &= 100 \times .05 \times 2^{1/2} = \\
 & \quad 7.07 \text{ g} \\
 \\
 \text{LEMUF} &= 2((4 \times 18^2) + (32 \times 2.35^2) + (120 \times .6^2) + 7.07^2)^{1/2} \\
 &= 79.1
 \end{aligned}$$

6.4.2 Independence of the variables measured in this model results in zero covariance.

6.5 Performance:

- 6.5.1 Measurement quality control is achieved through having all radiometric assays (6.2.1 and 6.2.3) made by a Quality Control group independent of the processing plant organization. Spectrometry equipment is calibrated at intervals with N.B.S. ^{235}U standards in the same form and enrichment as process solution samples. Volume calibrations are performed by weighing on balances standardized with standard weights. In the ND assay of finished items, standards in the form of finished items are used to assure equipment performance. These standardized items have known ^{235}U content, previously determined and referred back to the NBS standards.
- 6.5.2 An annual audit, by an individual appointed by the Nuclear Safeguards Committee, of MUF and LEMUF records will be performed to monitor and control performances.
- 6.5.3 Systematic errors will be reassessed through acquisition periodically of N.B.S. standards and through annual calibration of balances with standard weights by an independent organization. Random errors will be assessed at each inventory period when replicate determinations are made.

6.6 Verification: Computations of MUF and LEMUF will be double-checked by the two individuals assigned to complete each inventory (Section 5.3), or their designees.



PLANT PROCESS FLOW DIAGRAM

(Each block in flow chart represents a material balance area.)

APPENDIX D
TYPICAL
INVENTORY REPORT
 PERIOD ENDING _____

UO₂ FEED MBA

<u>Batch #</u>	<u>Batch Volume</u>	<u>Sample #</u>	<u>Assay Report</u>	<u>Bulk Determination</u>
Subtotal ²³⁵ U			_____	_____

PLATING, ENCAPSULATION, QUALITY CONTROL MBA

<u>Plating Solution #</u>	<u>Batch Volume</u>	<u>Sample #</u>	<u>Assay Report</u>	<u>Bulk Determination</u>
Subtotal ²³⁵ U			_____	_____

<u>Waste Solution #</u>	<u>Batch Volume</u>	<u>Sample #</u>		
Subtotal ²³⁵ U			_____	_____

<u>Target Serial #</u>	<u>Assay Report</u>			
Subtotal ²³⁵ U			_____	_____

APPENDIX D, CONT'D.

REACTOR OPERATIONS MBA

<u>Target Serial #</u>	<u>Assay Report</u>
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Subtotal ²³⁵U _____

RADIOCHEMICAL PROCESS MBA

<u>Batch #</u>	<u>Target Serial #</u>	<u>Assay Report</u>
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Subtotal ²³⁵U _____

MISCELLANEOUS

<u>Item</u>	<u>Assay</u>	<u>Basis of Assay</u>
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Subtotal ²³⁵U _____

Material Balance Total =====

APPENDIX D, CONT'D.

$$\text{LEMUF} = 2 \left[\frac{(M_s \times \sigma_s)^2}{N_s} + \frac{(M_x \times \sigma_x)^2}{N_x} + \frac{(M_t \times \sigma_t)^2}{N_t} + \frac{(M_w \times \sigma_w)^2}{N_w} \right]^{1/2}$$

M_s = SNM thru-put in UO_2 feed MBA.

N_s = # SNM batches received from supplier.

σ_s = Fractional standard error for UO_2 feed measurement.

M_x = SNM in solution transferred from UO_2 feed to plating encapsulation and QC (PEQ) MBA.

N_x = # of SNM batches transferred from UO_2 feed to PEQ MBA.

σ_x = Fractional standard error for feed transfer.

M_t = SNM in irradiation targets transferred from PEQ to reactor operations.

N_t = # of targets.

σ_t = Fractional standard error for target assay.

M_w = SNM in waste solution from PEQ to disposal.

N_w = # waste batches.

σ_w = Fractional standard error for waste solution from PEQ.

$$\text{MUF} = \text{BI} + \text{A} - \text{EI} - \text{R}$$

BI = Beginning Inventory.

A = Additions.

EI = Ending Inventory.

R = Removals.