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Division of Safeguards
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Gentlemen:

Enclosed please find the proposed Special Nuclear Material Quality Assurance Program for the Union Carbide Sterling Forest Laboratory. It has been prepared in accordance with your outline of 8/27/75 to be included as Chapter 4 of the Union Carbide Sterling Forest Research Laboratory Fundamental Nuclear Materials Control Plan.

Very truly yours,

James J. McGovern
Manager
Radiochemical Production

JJMcG:js
Enclosures (10)

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CHAPTER 4

4.0 MEASUREMENT QUALITY ASSURANCE CONTROL PROGRAM (MQA)

4.1 Organization and Management

4.1.1 Functional Assignment

The MQA program is the responsibility of the SNM Measurement Quality Assurance Supervisor. This position in the organization is shown in Figure "B" (Rev. 1 Enclosed)

The qualifications for this position shall be as follows:

1. Good knowledge of all the measurement systems used for SNM accounting.

2. Good knowledge of the statistical methods used to evaluate measurement accuracy.

3. Good knowledge of the license and regulatory requirements for SNM control and accounting.

4.1.2 Procedures

The SNM MQA supervisor, in conjunction with all operations managers, will initially write and maintain a MQA program procedures manual in accordance with the requirements of this section. This manual and subsequent modifications thereto will be reviewed and approved by the Nuclear Safeguards Committee (NSC). The manual will be reviewed annually by the NSC designated auditor.

4.1.3 Management Review

The MQA program will be audited by the SNM MQA supervisor every material balance period. His report will coincide with the inventory and LEMUF reports.

4.1.3 Internal Audits

The NSC thru a designee will audit this program annually.

4.1.5 Contractor Program Audit

Currently there is no vendor used for SNM measurement at the Sterling Forest site. If outside services are used in the future, the MQA supervisor shall require any vendor to establish an MQA program and he will audit the vendor's program annually.

4.2.1.1 Calibration and Standards

The standards which will be used as working and calibration standards in the U and U^{235} measurement program at the Sterling Forest Laboratory are as follows:

a. Highly enriched uranium ($\sim 93\%$ U^{235}) such as NBS-930 reference material or equivalent will be dissolved and made into concentrations, masses and geometries that are typical of the process materials. These items will be used as calibration and working standards for measuring U^{235} in process solutions.

b. Uranium of natural isotopic abundance such as NBS SNM 950 or 960 or equivalent will be dissolved to various concentrations that are typical of the process materials. These solutions will be used as calibration and working standards for measuring uranium in process solutions.

c. Sealed target tubes containing highly enriched uranium ($\sim 93\%$ U^{235}) shall be used as calibration and working standards for measuring U^{235} in process irradiation targets. These standards will span a range of target weights that are routinely used.

d. Class S analytical weights shall be used as calibration standards for volumetric vessels which are used in the analytical procedures.

e. A Class A volumetric flask (1 liter T.D.) will be used as a calibration standard for the large volumetric measurement vessels for the plating process solutions and waste solutions.

f. A Class A calibrated graduated cylinder (1 liter T.D.) will be used as a standard for small volumetric measuring vessels which are used to measure small volumes of process and waste solutions.

4.2.1.2 Certification

All standard solutions will be certified for their $uranium^{235}$ and total uranium content by independent analytical groups. The Union Carbide Corporation neutron activation analysis laboratory (U and U^{235}), the Oak Ridge National Laboratory analytical division (U and U^{235}), and the Union Carbide analytical services laboratory (total U only).

Recertification of calibration standard solutions will be accomplished annually. Working standard solutions will be recertified annually or more frequently if it is indicated that their values have changed. The sealed target tubes will be certified thru a cooperative calibration program conducted by the Sterling Forest Neutron Activation Analysis Laboratory and ORNL. A minimum of 15 targets, whose uranium^{235} content span the normal range of process target weights, will be counted by a γ ray spectrum analyser to determine a photo-peak area for the 185 KeV gamma ray emitted by the decay of U^{235} in each target. All but three targets will be destructively analyzed for U^{235} by UCCSFRL and ORNL. These results will be used to construct a calibration curve for this measurement system. This calibration curve will be recertified annually. The weights, and volumetric flasks will be certified by the manufacturer to have a certain accuracy and that the calibration of each is traceable to a national standard.

4.2.1.3 Traceability

Records on all calibration and working standards shall be kept so that each will be traceable to some national standard or natural physical constant.

4.2.1.4 Representativeness

a. The highly enriched U^{235} liquid standards, which will be made from the NBS-930 or equivalent material, will be similar in composition to the process materials being measured. The isotopic abundance of a standard shall be within 10% of that which is in the process solutions.

b. The uranium of natural isotopic abundance will be similar in uranium concentration to the process materials being measured.

c. The standard sealed targets will have uranium of comparable enrichment to that contained in the process targets. These standards will have weights of uranium which will bracket the normal weights of the process targets and they will be of the same size and materials as the process targets.

d. The Class "S" weights are representative of masses of solutions which are routinely used in small volumes.

e. The volumetric flasks will be representative of the typical volumes to be measured in the routine measurement of process samples.

4.2.1.5 Controls

The calibration standards will be in the custody of the SNM MQA Supervisor. They will be kept in a manner that will maintain their original characteristics. The working standards will be in the custody of the laboratory personnel performing the measurements. They will be maintained, as well as practicable, so that their original characteristics will not change.

4.2.2 Standard Measurements

1. Target Tube

A working standard shall be measured each time a group of process targets are to be assayed. The schedule for measuring the standard is dictated by production requirements, however; standards will be measured a minimum of once per material balance interval.

Approximately 5 technicians could be associated with these standard measurements.

2. Delayed Neutron

A working standard shall be measured each time a group of process samples are to be assayed. The schedule for measuring the standard is dictated by production requirements, however; working standards will be measured a minimum of once per material balance interval. The standard and routine process material measurements will coincide in the following areas:

1. Similar glassware.
2. Same micro pipeting apparatus.
3. Same method and type of encapsulation.
4. Same irradiation time, decay time, and counting time.
5. Comparable concentrations in solution.
6. Same neutron detector system.

Approximately 4 technicians could be associated with these standard measurements.

3. Total Uranium

A working standard shall be measured each time a process material sample is to be measured. The schedule for measuring the standard is dictated by production requirements, however; standards will be measured a minimum of once per material balance interval. The standard and process materials measurements will coincide in the following areas:

1. Same chemical procedure.
2. Same reagents.
3. Same original concentration.

Approximately 3 technicians could be associated with these standard measurements.

4. Paper Waste (U^{235} Gross Counting)

A working standard shall be measured each time a drum of waste material is to be measured. The schedule for measuring the standard is dictated by production requirements, however; standards will be measured a minimum of once per material balance interval. The standard and process waste material measurements will coincide in the following areas:

1. Same number of determinations per assay.
2. Same counting system.
3. Same geometry.

Sufficient counts will be made on the standard and process waste drums to compensate for differences in geometry.

Approximately 3 technicians could be associated with these standard measurements.

4.2.3 Calibration Systems

1. Calibration of Delayed Neutron System for Measurement of U^{235}

The initial calibration of the system will be performed by running at least 5 samples of 2 solutions, each made up from material of known isotopic abundance supplied by NBS (Item A, Section 4.2.1.1). Calibration standards and working standard solutions will be made from this material. This calibration will be done over a range of 1 to 50 micrograms of U^{235} per sample, in order to bracket the range of U^{235} values which are normally found in samples submitted for assay. Recalibration of working standards shall be performed at least every 2 months, whenever a new working standard solution is introduced into the assay scheme, or when the value determined for the U^{235} content of the working standard falls outside the 95% confidence level on the control chart on 2 of 3 analyses.

2. Calibration of System for Radiometric Determination of U^{235} in Target Tubes

The initial calibration of this system will be performed by counting each of 15 target tubes at least 8 times, rotating and/or revolving the tubes after each count to minimize variations due to distribution of the uranium plate. Following these counts, 12 of the tubes will be opened, and the uranium plating will be stripped from them. The resulting solutions will be analyzed for both total U and U^{235} in comparison with standard solutions of known total U and U^{235} content (Item a., Section 4.2.1.1). The results of these analyses will be used to determine a value of counts per minute per gram of U^{235} in the target tubes. The 15 tubes used for this calibration will contain amounts of U^{235} which span the full range of process target weights. In addition, the three tubes not opened and stripped of uranium will be chosen on the basis of U^{235} content (one of ~ 8 grams, one of ~ 11 grams, and one of ~ 15 grams) and they will be retained to be used as calibration standards (Item c., Section 4.2.1.1) Recalibration over the entire range of useable target weights will be performed at least once a year. A minimum of one target will be sacrificed (i.e., counted, stripped of uranium, and the resulting solution being assayed) during each inventory period (2 months). In addition, a recalibration, using Item c., Section 4.2.1.1, will be performed whenever the results of the destructive analysis of a target disagree with the radiometric analysis by more than 5%.

3. Calibration of System for Determination of Total U by Titration

The initial calibration of this system will be performed by analyzing, in triplicate, samples of solutions whose composition and uranium content is representative of the production samples to be analyzed, including Item b., Section 4.2.1.1. Portions of the same solutions will be submitted to ORNL for analysis. This procedure will be followed until the results of our analysis and that performed by ORNL agree to within one standard deviation of each set (i.e., until the spread of results on our analysis and that performed by ORNL overlap). Once this agreement has been reached, the system will be recalibrated by the same procedure at least once per year. Working standards which are made from Item b., Section 4.2.1.1, will be analyzed at least every 2 months to ensure that the system remains calibrated.

Recalibration of working standards will be performed whenever the assay results stray beyond the limits determined at the 95% confidence level determined at the calibration of the solution.

4. Calibration of Volume Measurements

Whenever possible, initial calibration of this system will be performed by direct comparison to the national system of measurements through the use of available standardized and certified vessels. In cases where this is impractical, initial calibration will be performed by comparison of the mass of the delivered or contained volume of solution (whichever is applicable) with the mass of standardized weights traceable to the national system, with appropriate correction for temperature and specific gravity. This calibration shall be performed on all vessels which are used in the process. The standards to be used are identified in Section 4.2.1.1 as Item d., e., and f. Recalibration of vessels shall be performed at least once per year, whenever new vessels are introduced into the system, or whenever there exists sufficient discrepancy in the results of analyses to cause the calibration to be suspect.

5. Calibration of System for Determining U^{235} Content in Drums Containing Solid Waste.

The system will initally be calibrated by suspending vessels containing known amounts of U^{235} within a drum typical of those used for disposing of solid waste. These vessels will contain quantities of U^{235} spanning the range which could be shipped as solid waste in a drum (0-15 grams). At least 15 analyses will be performed with each vessel positioned at various positions within the drum, including those positions where the maximum count rate (i.e., vessel as close as possible to the detector), minimum count rate (i.e., vessel as far from detector as possible) and intermediate count rates will be encountered. From this data, an average value of counts per minute per gram of U^{235} in the drum will be determined, along with limits of error for this type of analysis. The calibration standards used for this system will consist of accurately dispensed amounts of the U^{235} standard described in 4.2.1.1. A working standard, consisting of a sealed drum within which a known amount of U^{235} has been randomly distributed, will also be fabricated and analyses performed on it in conjunction with those done on waste drums. Recalibration of the system will be performed at least once per year, or whenever determinations performed on the working standard do not fall within acceptable (95%) confidence limits.

4.2.4 Statistics

Since the calibration of the measurement systems will be an ongoing process performed in conjunction with the analyses necessary for SNM control, continuous statistical evaluation of the standard measurements will be carried out. A 95% confidence level will be assigned to all standard measurements and control charts will be maintained on these measurements.

The systematic error variance will be calculated as follows:

Systematic error variance $= \beta^2 + So^2$

where $\beta = (\bar{x} - Uo)$

So^2 = Variance describing the uncertianty of Uo

Uo = Absolute value assigned to the standard

$\bar{x}$ = Average of all measurements on a particular standard.

(Reference Jaech, Statistical Methods in Nuclear Material Control, Section 3.3.2, pp. 88 - 90)

Since it is intended to maintain all standard measurements within 2σ of the original calibration measurements, and it is not intended to make small bias corrections, it is considered appropriate to assign this bias as the systematic error variance.

4.3 Sampling Accuracy

4.3.1 Sampling for the measurement of U^{235} in solutions by the delayed neutron method of analysis will be accomplished as follows:

a. The solution shall be stirred thoroughly just prior to the bulk tap.

b. Three bulk taps will be removed and placed into clean glass bottles which will be covered for transport to the analytical lab. The samples will be drawn with a long tube which will be submerged to the entire depth of the bulk solution so that all layers in the bulk solution will be sampled.

c. Two bulk taps will be agitated thoroughly and one sample will be taken of each. These samples will be analyzed for U^{235} content.

d. In the event the two samples from the two bulk taps disagree by more than 10%, one sample will be drawn from the 3rd bulk tap for analysis.

This procedure is considered adequate for maintaining continuous control on sampling accuracy.

4.3.2 Sampling for the measurement of total uranium in solutions by the chemical titration method of analysis will be accomplished in the same manner as outlined in 4.3.1.

NOTE: Due to the radiation exposure hazard associated with the radioactive waste solutions only one bulk tap will be drawn and one sample will be analyzed.

4.3.3 There will not be an error contribution due to sampling in the assay of targets and waste drums since the whole item is assayed in each case.

4.4 <u>Measurement Precision</u>

4.4.1 <u>Program Description</u>

The chemical analytical procedure for uranium analysis and the delayed neutron procedure for U^{235} analysis are as follows:

1. Measuring the volume of the material to be analyzed.

2. Taking bulk taps (3 each).

3. Taking a measured volume from the bulk taps and analyzing them (2 analyses; 1 from each of 2 bulk taps; a spare bulk tap is to be held in reserve).

The gamma ray spectroscopy procedure for analyzing sealed targets involves counting each target in a reproduciable geometry for 5 to 10 minutes and computing a photo peak area for the 185 KeV γ ray. Working standard target tubes will be counted along with process targets at least 3 times.

The gamma ray spectroscopy procedure for analyzing waste drums will be to count a standard drum at 5 equally spaced locations along 3 horizontal planes at the bottom, middle and top of each drum. Drums containing the process waste will be counted in an identical manner.

The number of process measurements has been dictated by production schedules. To date the schedule has been to perform a full set of process measurements on the average of once per week. (A full set of measurements means, 4 plating solutions, 4 spent plating solutions, 1 plating waste solution, one radioactive waste solution, 16 targets and one solid waste drum.) A minimum number of one set of replicate samples will be analyzed each material balance period if there were no production operations with SNM. All measurements on process materials shall be performed concurrently with measurements on working standards. The measurements of these standards will be representatively spread across all operators and areas throughout each material balance period so that an average of all variance in such a period will account for between-operator, between equipment, etc.... caused variances.

4.4.2. Statistics

The replicate data from the working standard measurements shall be used to derive variances and standard deviations for random error calculations. Representative sets of data for each measurement system (measurement system includes sampling and analysis) throughout each material balance period will be chosen. The variance and relative standard deviation for each set of data will be calculated. The average of all standard deviations for each measurement system on each material type will be used for calculating the LEMUF at the end of each material balance period. (Reference Jaech, Statistical Methods in Nuclear Material Control, Section 3.3.2, pp. 88 - 90.)

4.5 Control Program

4.5.1 Refer to Section 4.3. The program of replicate sampling and the continuous evaluation of the measurement results relative to preset limits is considered to be sufficient control on all sampling for analysis.

Refer to Section 4.4. The program for continuous analysis of working standards and the evaluation of the results of these analyses relative to limits at a 95% confidence level is considered to be sufficient control on all of the measurement systems. Control charts based on calibration standards measurement data will reflect the 95% confidence level.

4.5.2 Control Limits

Control limits will be established on control charts (95% confidence level) using data from the measurement of calibration standards. The current control data will be evaluated for purposes of updating the control limits on a bi-monthly basis. Control limits will be updated at least once a year or in any case, when determinations on working standards fall outside the 95% confidence level on 2 of 3 replicate analyses.

4.5.3 Control Response

When sampling or measurement performance falls outside of control limits the data will be disregarded and the system will be recalibrated using reference calibration standards.

4.6 Records and Reports

The details of the periodic calibration of all measurement operations (volume, sampling and analysis) shall be maintained by the Measurement Quality Assurance Supervisor. These records will include the results of each measurement that is made and the details of the calculations for constructing control charts.

The summaries of error data used in LEMUF calculations will be kept by the SNM Accountability Officer.

Statistical control records in the form of control charts will be kept and maintained by the analytical people performing the respective measurements.

Reports of corrective actions shall be kept by the SNM Accountability Officer.

Report of the SNM QA measurement review during each material balance period will be maintained by the Measurement Quality Assurance Supervisor.

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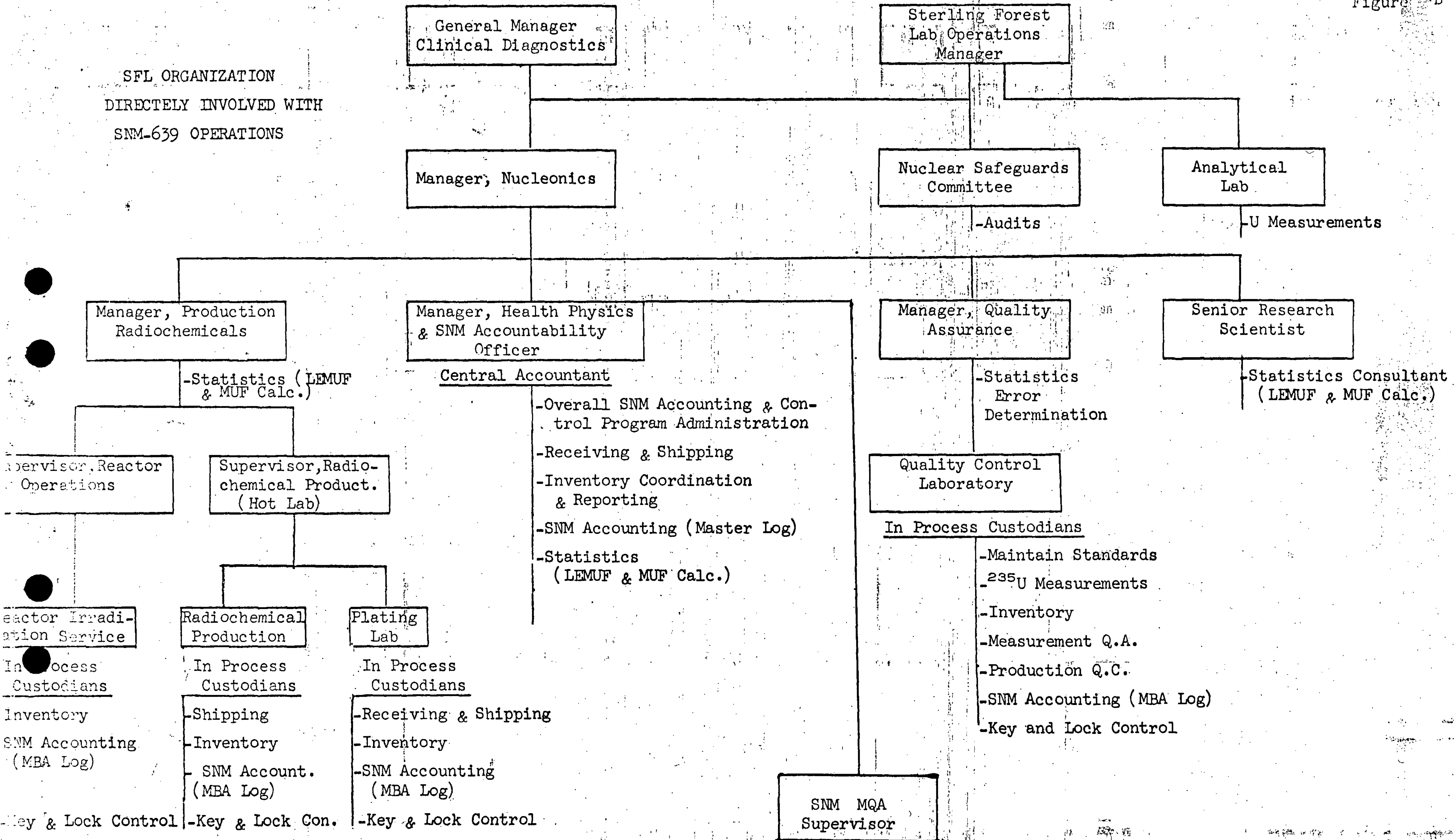

Figure B, Rev. 1