Regulatory Docket File

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UNION CARBIDE

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June 23, 1978

U. S. Nuclear Regulatory Commission
Material Control & Licensing Branch
Division of Safeguards
Washington, D. C. 20555

Attn: Mr. James G. Partlow, Chief

Dear Sir:

We have enclosed a completely revised edition of the Union Carbide Corporation (UCC) Measurement Quality Assurance Program (Chapter (4) of our FNMC Plan). It has been rewritten to include changes that were made as a result of your comments on our submission of 5/30/78.

This supersedes entirely our submission of 5/30/78.

Very truly yours,

James J. McGovern
Manager
Radiochemical Production

JJMcG:js
Enclosures (10)

FEE EXEMPT
(70.57)

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 Manager of Quality Assurance. The duties of Accountability Coordinator have been assigned to the Manager of Health, Safety and Environmental Affairs and those of Control Coordinator to our Health Physics Supervisor. From the organizational chart enclosed as Figure "B" it can be seen that there is separation of function between the Accountability Coordinator and the Quality Assurance organization. The Measurement Control Coordinator reports directly to the Accountability Coordinator who in turn reports to the Site Operations Manager.

The qualifications of both the Accountability Coordinator and the Measurement Control Coordinator (MCC) include the following:

a) Working knowledge of all the measurements systems used for SNM accounting.

b) Working knowledge of the statistical methods used to evaluate measurement accuracy.

c) Working knowledge of the license and regulatory requirements for SNM control and accounting.

d) Bachelor's degree or equivalent job experience (4-6 years) in related work.

4.1.2 Procedures

The Manager of Quality Assurance will be responsible for preparing and maintaining the measurements quality assurance manual. This manual and subsequent modifications shall be reviewed and approved by both the Accountability Coordinator and the Measurement Control Coordinator. The Measurement Control Coordinator shall review the entire manual at least annually.

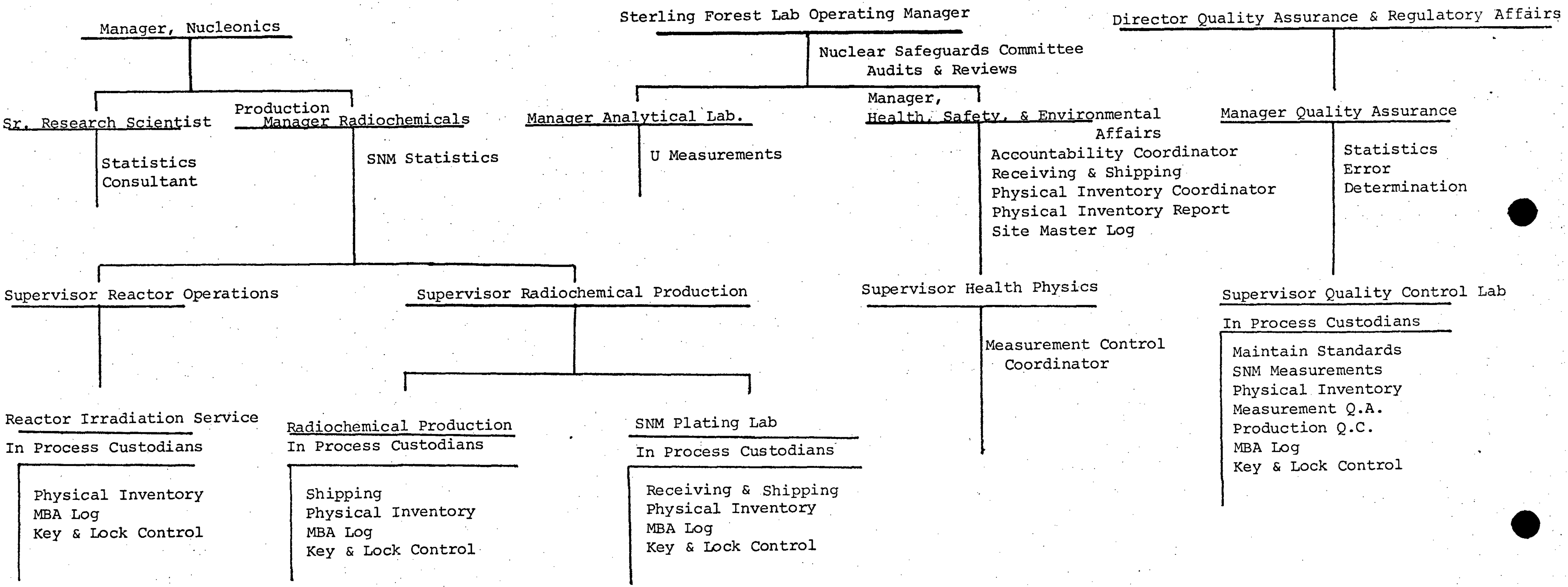

FIGURE B

STERLING FOREST ORGANIZATION

INVOLVED WITH SNM

4.1.2 Procedures (cont'd)

The manual shall include:

a) Providing and maintaining reference standards.

b) Calibrating measurement systems.

c) Performing bulk measurements.

d) Obtaining samples.

e) Recording program data.

f) Controlling measurement performance.

4.1.3 Management Review

At least annually, the Nuclear Safeguards Committee shall designate a technically competent individual or group of individuals to review the management of the measurement quality assurance program. This review shall be structured to determine whether the effectiveness of the program is being maintained. Reviewers shall not be directly involved in the area they are reviewing. They will be neither the Accountability Coordinator nor the Measurement Contorl Coordinator. A review team may consist of members who are responsible for functions with MBA's however, they may not review their own functions and there will be no cross review of functions.

A written report of this review shall be submitted to the Nuclear Safeguards Committee, the Measurement Control Coordinator and the Accountability Coordinator.

4.1.4 Internal Audits

At least annually the Nuclear Safeguards Committee shall designate a technically competent individual or group of individuals to audit compliance with the measurement quality assurance procedures. Auditors shall not be directly involved in the area they are auditing, and there will be no cross audit of functions.

A written report of this audit shall be submitted to the Nuclear Safeguards Committee, the Measurement Control Coordinator and the Accountability Coordinator.

4.1.5 Contractor Program Audits

No measurement services are provided by outside contractors or laboratories.

4.2 CALIBRATION AND STANDARDS

4.2.1 Reference Standards

A. Primary standards will consist of material obtained from National Bureau of Standards or New Brunswick Laboratories.

a) Natural Uranium Primary Standards will be made by the Analytical Department from certified U_3O_8 or equivalent material dissolved in acid to form a solution of uranyl nitrate. This material will be used to make standards to verify total uranium assay procedures.

b) Enriched Uranium Primary Standards will be made by the Analytical Department from National Bureau of Standards or New Brunswick Laboratories enriched standard material whose U-235 enrichment most closely approximates the enriched material currently being used. It will be made into uranyl nitrate solution to verify U-235 assay procedures and calibrate working standard material.

c) A set of Class S weights, 10 mg - 100 gms, will be used to calibrate laboratory analytical balances.

d) Purchased, individually certificated, Class A volumetric flasks will be used to prepare and hold liquid primary standards.

B. Calibration and Working Standards will be purchased certified material or material calibrated against Primary Standards or Primary Standard Material.

a) Cs-137 point source, calibrated by National Bureau of Standards, shall be used to calibrate and monitor multichannel analyzers and detectors.

4.2.1 Reference Standards (cont'd)

b) Sealed ampoules containing known quantities of U-235 in solution, shall be used to calibrate and monitor multichannel analyzers. These solutions will be made from Primary Standard Material.

c) Natural uranium standards for total uranium will be made from reagent grade uranyl nitrate in concentrations that span the range of total uranium that is normally encountered in test solutions. This material will be calibrated against the primary standard material when it is made up by the Analytical Department.

d) Enriched U-235 working standard material will be made from actual feed material and calibrated against primary U-235 standard material.

e) Sealed target tubes for non-destructive analysis tests containing enriched stock uranium shall be used both as Calibration and Working Standards. These tubes shall be calibrated against similar target tubes which were NDA tested, stripped and calibrated against Primary Standard Material.

f) Standard Waste Barrels used for NDA testing will be prepared for use as both Calibration and Working Standards. The waste barrels will be prepared with liquid enriched solution calibrated against Primary Standards and containing the solution absorbed on paper in random configurations.

4.2.1.1 Current List Of All Reference 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) Enriched uranium (93% U-235) primary standard solution for calibration of working standards.

b) Natural Uranium Primary Standard solution to calibrate working standards.

c) Set of Class S weights to calibrate laboratory balances.

4.2.1.1 Current List Of All Reference Standards (cont'd)

d) Cs-137 NBS point source to calibrate multichannel analyzers.

e) Uranium-235 solution in sealed ampoules to calibrate multichannel analyzers.

f) Solutions of natural uranium of at least two different strengths to encompass the strengths of process solutions.

g) Enriched U-235 working standard solution.

h) Standard Waste Barrels to calibrate waste barrel measurements.

i) Sealed target tubes containing enriched Uranium (~93% U-235) to calibrate irradiation target measurements. These standard tubes will span the range of target weights that are normally encountered.

j) Class A certified volumetric flasks (1 liter T.D.) to calibrate volumetric measurement vessels that are used for the plating process solutions and waste solutions.

k) Class A certified glass pipettes will be used for analytical and quality control work involving total uranium measurements.

4.2.1.2 Certification

All primary standard solutions will be certified for uranium-235 or total uranium content by independent analytical groups and directly traceable to a National Standard. All other working standards will be calibrated to the primary standard. Other purchased standard material will have vendor certified values traceable to a National Standard.

a) Uranium-235 Primary Standard solution is made from a weighed known amount of certified NBS or equivalent material and is verified by analysis. Initial standards will be verified by independent analytical groups. Subsequent standards will be verified by comparison to current standards.

4.2.1.2 Certification (cont'd)

b) Natural Uranium Primary Standard solution is made from weighed known amount of certified NBS or equivalent material and is verified by analysis (as in (a) above).

c) Class S weights will be purchased with a certificate traceable to a National Standard.

d) Cs-137 is a purchased NBS certified point source.

e) Uranium-235 solutions of various concentrations of material from (a) above are sealed in ampoules. These are non-consumed standards unless accidentally broken. These ampoules will be verified by establishing a duplicate set of samples covering a range of concentrations. Counting data from these sets should fall along a continuous curve.

f) Natural uranium working solutions will be calibrated against the primary standard by the methods indicated in the assay procedures for special nuclear material by the Analytical Department.

g) Enriched U-235 working standard solutions will be calibrated against the Uranium-235 Primary Standard solution by the radiometric test in the Assay Procedures for SNM by the Quality Control Department.

h) The standard waste barrels will be prepared using working standard Uranium-235 solutions calibrated as in (g) above and certified by the Quality Control Department.

i) Sealed target tubes will be gathered at random covering the range of Uranium-235 loaded that is normally encountered. The tubes will be counted in the procedure outlined under Assay Procedures for SNM. Once counted, the tube contents will be dissolved into a known volume and the solutions will be assayed against the enriched working standards as in (g) above. The results will be plotted against the NDA count rate obtained from counting each tube.

4.2.1.2 Certification (cont'd)

j) The Class A volumetric flasks will be purchased with certificates of traceability. (Each vessel will be individually certified.)

k) The Class A glass pipettes will be purchased with individual certificates of traceability. Calibration will be verified by measuring volumes of water and weighing against Class S weights.

Recertification of Working Standard Solutions will be accomplished annually or more frequently if it is indicated that their values have changed. The sealed target tubes will be certified thru a calibration program conducted by the Sterling Forest R&D Department and the Quality Control Department. A minimum of 8 targets, whose Uranium-235 content span the normal range of process target weights, will be counted by a γ ray spectrum analyzer 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. These results will be used to recertify the calibration curve for this measurement system.

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.

The method of traceability of each standard to a national standard has been described in Section 4.2.1.2. Each standard is listed below, showing which National Standard it will be traceable to.

a) Uranium-235 Primary Standard traceable to NBS or NBL standards.

b) Natural Uraninum Primary Standard traceable to NBS or NBL standards.

c) Class S weights traceable to NBS.

d) Cs-137 calibration point traceable to NBS.

e) Uranium-235 solution in a sealed ampoule traceable to NBS or NBL standards.

4.2.1.3 Traceability (cont'd)

f) Natural Uranium working standard traceable to NBS or NBL standards.

g) Uranium-235 working standards traceable to NBS or NBL standards.

h) The standard Waste Barrels are traceable to NBS or NBL standards.

i) The Uranium-235 content of the Sealed Target Tube standards is traceable to NBS or NBL standards.

j) The Class A Volumetric Flasks are traceable to NBS standards.

k) The Class A Glass Pipettes are traceable to NBS standards.

4.2.1.4 Representativeness

Each uranium standard shall be representative of the composition and enrichment of the process material being measured.

a) The enriched U-235 liquid standards will be made from the NBS-930 or equivalent material and 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 used.

4.2.1.4 Representativeness (cont'd)

e) The Class A volumetric flasks will be representative of the typical volumes to be measured in the routine measurement of process samples and in the make-up of standards.

f) The Standard Waste Barrel will be made with waste laboratory material that has been doped with calibrated uranium reference material.

4.2.1.5 Controls

A representative portion of each liquid and sealed target calibration standard will be in the custody of the MCC. They will be kept in a manner that will maintain their original characteristics. The working standards and portions of the Primary Standards for laboratory use will be in the custody of the laboratory personnel performing the measurements. They will be maintained so that their original characteristics will not change. Because Working Standard solutions and Primary Standard solutions will be used over several months, they will be packaged into small volumes to be used during a 2-3 week period. Vials, flasks and bottles will be sealed in such a way as to minimize spillage or evaporation.

One vial, flask or bottle of each solution type will be given to the MCC to be held in a locked cabinet. The remaining vials, flasks or bottles will be held in the Quality Control Laboratory.

Representative sealed target tubes covering the weight range of use will be held by the MCC in a locked cabinet.

The Class S weights will be held by the Manager of Pharmaceutical Quality Assurance in his office.

The Class A flasks and pipettes will be held by each respective laboratory.

There will be no reserve Waste Barrel Standard held by the MCC.

4.2.2 Standard Measurements

Working Standards and/or Primary Standards will be measured as appropriate each time an analysis is performed. Values obtained for the standards will be recorded on control charts to assure the operating technician that the obtained value is within limits. If the value is outside control chart limits, the analysis will be repeated. If still outside of limits, the technician is to stop and report the incident to his line Supervisor or Quality Assurance Manager. (see Section 4.3)

During a material balance period, appropriate standards shall be measured a minimum of 16 times for each measurement system being used (based on two standards measurements per week).

a) Target Tube (Radiometric Method)

A working standard shall be measured each time a group of process targets are to be assayed.

Approximately 5 technicians could be associated with these measurements.

b) Total Uranium (Chemical Method)

A working standard shall be measured each time a process material sample is to be measured. 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 measurements.

c) Uranium-235 (Radiometric Method)

A working standard (flame sealed ampoule) will be measured each time a process material sample is to be measured. The standard and the process material will be measured on the same detector and multichannel analyzer in succession.

Approximately 5 technicians could be associated with these measurements.

4.2.2 Standard Measurements (cont'd)

d) Uranium-235 (Back-up Method Using Delayed Neutrons)

A working standard shall be measured each time a group of process samples are to be assayed. The standard and the process material measurements will coincide in the following areas:

1. Similar glassware.

2. Same micro pipetting 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 5 technicians could be associated with these measurements.

e) Cs-137 (Calibration Standard)

This sealed point source standard will be measured each time the multichannel analyzer system is used for analysis. The values obtained will be recorded on control charts.

Approximately 5 technicians could be associated with these measurements.

f) Paper Waste (U-235 Gross Counting)

A working standard barrel shall be measured each time a drum of waste material is to be measured. 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 possible differences in geometry.

Approximately 5 technicians could be associated with these measurements.

4.2.3 Calibration Systems

The initial calibration runs have been completed and results of these calibrations are found in the Assay Procedures for Special Nuclear Materials.

Each time a new detector system is installed a recalibration of the system will be completed. Revalidation of each system will be made whenever control charts indicate a problem.

a) Calibration of System for Radiometric Determination of U-235 in Target Tubes

The initial calibration of this system was 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 were opened, and the uranium plating stripped from them. The resulting solutions analyzed for both total U and U-235 in comparison with standard solutions of known total U and U-235 content. The results of these analyses were used to determine a value of counts per minute per gram of U-235 in the target tubes. The 15 tubes that were used for this calibration contained amounts of U-235 which span the full range of process target weights. Three tubes, not opened and stripped of uranium, were chosen on the basis of U-235 content (one of ~8 grams, one of ~ 11 grams, and one of ~15 grams) and they were retained to be used as calibration standards. Recalibration over the entire range of useable target weights will be performed at least once every 2 years. A minimum of one target will be sacrificed (i.e., counted, stripped of uranium, and the resulting solution being assayed) during each two inventory periods (4 months). In addition, a recalibration will be performed whenever the results of the destructive analysis of a target disagrees with the radiometric analysis by more than 2 σ of destructive analysis results.

b) Calibration of System for Determination of Total U by Titration

The initial calibration of this system was performed by analyzing, in triplicate, samples of solutions whose composition and uranium content was representative of the production samples to be analyzed.

4.2.3 Calibration System (cont'd)

Portions of the same solutions were submitted to ORNL for analysis. This procedure was followed until the results of our analysis and that performed by ORNL agreed to within one standard deviation of each set (i.e., until the spread of results on our analysis and that performed by ORNL overlap.)

The system will be recalibrated by the same procedure at least once per year. Working standards will be analyzed during each analysis of process material. The working standard will be similar in concentration of uranium in the process sample.

Recalibration of working standards will be performed whenever the assay results stray beyond the limits determined at the 95% confidence level as derived at the original certification.

c) Calibration of System for Determination of Total Uranium-235 in Solution by Radiometric Assay

The initial calibration of this sytem was performed by analyzing, in duplicate, varying amounts (total grams of uranium-235) in a constant volume of solution. This was done by pipetting different quantities of the Primary Standard solution of Uranium-235 and diluting each to a constant volume. These represented a range of concentrations of SNM in solution typical of process materials. These samples were counted (4 times each) to establish the instrument calibration (cts/5 min./gm.) for U-235 contained in solution. At the same time, flame sealed ampoules of the Uranium-235 solution were made to be used as non-destructive working calibration standards to be counted with each assay to assure the system continued operability within control chart limits. Each sealed ampoule was counted 10 times to derive the calibration constant. This calibration procedure will be repeated each time a new detector or analyzer is installed for uranium-235 analysis.

4.2.3 Calibration Systems (cont'd)

d) Calibration of Delayed Neutron System for Measurement of U-235 (Back-up Method)

The initial calibration of the system was performed by running at least 5 samples each of 2 solutions (10 total), each made up from material of known isotopic abundance supplied by NBS. Calibration standards and working standard solutions were made from this material. This calibration was 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 4 months, or when 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. If this assay method is not used during an M.B. period it will not be calibrated.

e) Calibration of System for Determining U-235 Content in Drums Containing Solid Waste

The system will be calibrated initially by suspending vessels containing known amounts of U-235 within a drum that is 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 these vessels located 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 consisted of accurately dispensed amounts of the U-235 working standard. A working standard drum, within which a known amount of U-235 has been randomly distributed, will be fabricated and analyzed in conjunction with waste drums. Recalibration of the system will be performed at least once per year, or whenever analyses of the working standard does not fall within acceptable (95%) confidence limits.

4.2.3 Calibration Systems (cont'd)

f) 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. Recalibration of vessels shall be performed at least once per year, whenever new vessels are introduced into the system, or whenever there is sufficient discrepancy in the results of analysis to cause the calibration to be suspect.

g) Calibration of Non-Certified Quality Control Pipettes

Because these pipettes (Eppendorff-10 - 1000λ) are not certified, they will be calibrated on a 6 month interval delivering the rated volume of water into a weighing boat and weighing the amount of water delivered. A minimum of twn measurements will be made for each pipette. If the values obtained show the pipette to be biased high or low by more than 3%, that pipette will not be used.

h) Balances shall be calibrated annually by weighing Class S weights. The Class S weights will be checked annually by an independent set of weights.

4.2.4 Statistics

4.2.4.1 Bias

Standards will be run before and after each group of process samples. Results of these measurements will be used to estimate the bias for each measurement system monthly. Plots of standards measurements every time process materials are measured should indicate if the current bias correction is valid. Bias corrections will not be required if either:

4.2.4.1 Bias (cont'd)

1. The estimated bias is less than:

   a) The uncertainty of the SRM included in the standards.

   b) The uncertainty on the WCTM assigned value (10% of σ of standards measurements during previous month).

or:

2. Data from the measurements of standards before and after the measurement of process materials is used to determine values for SNM content in lieu of prior calibration data.

4.2.4.2 Systematic Error

The uncertainty associated with the assigned value of each standard will be the systematic error for the respective measurement system and material types.

Each systematic error variance used in the LEMUF calculation will be the square of the uncertainty associated with a parameter rather than the square of the parameter.

4.3 SAMPLING ACCURACY

4.3.1 Sampling for U-235 Measurement in Solutions

Sampling for the measurement of U-235 in solutions by the delayed neutron or radiometric method of analysis will be accomplished as follows:

a) The solution shall be stirred thoroughly just prior to the bulk tap. Solids which may be present in feed solutions will be filtered. After the assay results confirm the shipper's assay, this material will be added to SNM waste and measured for SNM content along with other SNM waste present in the waste barrel.

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.

4.3.1 Sampling for U-235 Measurement in Solutions (cont'd)

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 5% (~ 95% confidence level), one sample will be drawn from the 3rd bulk tap for analysis.

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

4.3.2 Sampling for U Measurement in Solutions

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.

4.3.4 Monitoring Accuracy

Accuracy of sampling is monitored thru the analysis on two samples, one from each of two bulk taps, and comparing results so that each fall within 5% of the other.

4.3.5 Transport Integrity

All samples of liquid material are to be contained in closed vessels. Accurate samples for analysis are taken as a first step in analysis and there is no delay between sampling and analysis.

4.4 MEASUREMENT PRECISION

4.4.1 Program Description

The chemical analytical procedures for uranium analysis and the delayed neutron and radiometric procedures for U-235 analysis on feed, plating, waste and radioactive waste solutions 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 reproducible geometry for 5 to 10 minutes and computing a photo peak area for 185 KeV γ ray. Working Standard target tubes will be counted along with process targets at least 3 times.

The gamma ray spectroscopy (radiometric) 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.

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 variances in such a period will account for between-operator, between-equipment, etc...caused variances. A minimum of 16 measurements shall be made of each standard during the course of a material balance period.

4.4.2 Statistics

When a LEMUF calculation is required, data from the working standards and process material 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.

4.4.2 Statistics (cont'd)

The variance and relative standard deviation for each set of data will be calculated. The average of all standard deviations within each measurement system on each material type will be used for calculating the LEMUF (if required) and plotting control charts during each material balance period. (Reference Jaech, Statistical Methods in Nuclear Material Control, Section 3.3.2, pp. 88 - 90.)

Different material types exhibiting similar random error behavior may be grouped to derive a single variance value, provided:

1. The data is properly tested for normality.

2. The data from all types are utilized and,

3. The variance from the mixed types remains equal to that from a single type.

Normally, variances for each type of material will be calculated.

4.5 CONTROL PROGRAM

4.5.1 Basic Program

Results of analyses on working standards and process materials are plotted on control charts and evaluated continuously by laboratory personnel who do the measurements.

The program of replicate sampling and the continuous evaluation of the measurement results relative to preset limits is considered to be sufficient control on sampling in each measurement system. (See Section 4.3)

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 the measurement system. $\bar{x}$ & R control charts, based on calibration standards measurement data, will reflect the 95% and 99.9% confidence levels. (See Section 4.4)

4.5.1.1 If working standard measurements fall outside of the 95% confidence level limits, the measurement system will be investigated to determine the cause and corrective action will be taken to bring measurements back into the 95% confidence level limits.

4.5.1.2 If working standard measurements fall outside the 99.9% confidence level, no further measurements will be made until corrective action is completed to bring measurements into control.

4.5.1.3 The supervisor of the assay group shall review control charts bi-weekly to detect trends in the recorded data, out of control measurements and appropriateness of current limits.

4.5.1.4 The MCC, at the end of each mateial balance period, shall examine the recorded data for randomness, normality and out of control measurements. Results of this audit shall be reported to the Accountability Coordinator and the Manager Quality Assurance.

4.5.1.5 New control chart limits shall be calculated:

1. If current limits are not appropriate.
2. Previous data is not stable.
3. When WCTM is changed.

4.5.2 Control Limits

Data from the certification of WCTM shall be used to construct control charts. Control limits at the 95% & 99.9% confidence levels shall be approved by the MCC and they shall be plotted on control charts. ($\bar{x}$ & R charts). The current control data will be evaluated and updated as necessary. (See Section 4.5.1)

The average value assigned WCTM shall be the $\bar{x}$ value of control charts. Limits at the 95% & 99.9% levels shall be 1.96 σ & 3.29 σ respectively for $\bar{x}$ charts and 2.456 $\bar{R}$ & 4.122 $\bar{R}$ respectively for R charts.

4.5.3 Control Response

4.5.3.1 If measurement performance falls between the 95% & 99.9% confidence level control limits:

1. The MCC & Accountability Coordinator shall be informed.

2. The cause for out of control measurements shall be sought.

3. Records of actions shall be maintained.

4. MCC shall certify that the system is back in control (within 95% confidence limits).

4.5.3.2 If measurement performance falls outside of the 99.9% confidence level (any single measurement) control limits:

1. Measurements of this type of material shall not be used for accounting purposes until corrective action has been completed.

2. Follow procedures in (4.5.3.1) above.

4.6 RECORDS AND REPORTS

The Measurement Control Coordinator shall be responsible for the organization and storage of all records pertinent to the measurement quality control program. These records will be maintained so that access will be controlled by the Measurement Control Coordinator (MCC). The area will be chosen so as to allow prompt (24 hour) retrieval of information generated within the past 12 months. The information will be organized in such a fashion that each reported result is readily relatable to the original measurement, on the basis of lot number, item identification, date, or other correlation, as appropriate to the type of data. Such information will be retained for five years, with the exception of training and qualification records, which will be retained for two years. The following items are specifically included in this information if their use is required under other sections of the Measurement Quality Control Program:

Control Charts
Control Data Analyses
Calibration Data
Source Data of LE Calculations (when necessary)
Limit of Error Calculations (when necessary)
Measurement Results
Standard Preparation Data
Management Reviews
Audit Results
Corrective Action Reports
Contractor Reviews and Audits (if necessary)

4.6.1 (cont'd)

Sampling Accuracy Studies
Engineering Analysis and Evaluations
Normality and Randomness Tests
Efforts/Benefit Analyses

4.6.2 Statistical control records in the form of control charts will be kept and maintained by the Q.C. section responsible for the performance of the respective measurements while such control charts are currently in use. When these charts have been changed, outdated, or superseded, they will be transferred to the control of the MCC. In any case where a revision of a control chart is deemed appropriate by the Q.C. group, the MCC will be informed before such revision takes effect, so that he may approve any changes and see to it that they are properly recorded.

4.6.3 Control and calibration data as necessary under 4.2, 4.3, and 4.4 will be submitted to the MCC. With the assistance (if necessary) of other individuals fulfilling the requirements of 4.1.1.C-2, such analyses and tests as are appropriate under 4.5 will be conducted to monitor and control sampling and measurement performance. The results of these tests, along with their supporting data, will be stored as described in paragraph 1.

4.7 <u>TRAINING</u>

4.7.1 <u>Basic Program</u>

Due to the specialized nature of the processes performed under SNM-639, and the small scale of the operation and staff, training will generally be carried out using an "on-the-job" approach, through which inexperienced personnel will first be required to study the procedure for a particular measurement operation, then observe these sampling and/or measurement techniques as performed by an experienced analyst, and finally perform the tasks themselves under direct supervision. Such close observation of their performance will be continued until such time that sufficient data has been generated to demonstrate that the quality of the measurement being performed is

4.7.1 Basic Program (cont'd)

comparable to that of the same measurement as performed by experienced personnel. At this point, they will be considered to be trained in that procedure, and written documentation affirming their qualification to perform the procedure will be furnished to the MCC by the supervisor directly responsible for their training.

4.7.2 Extent of Training

Training of each operating technician will be appropriate to the role in the production or analysis sequence fulfilled by that individual and will be administrered by the supervisor of the area in which the function is performed. For example, production technicians will be instructed in good laboratory practice and the correct, precise use of balances and volume measuring devices so that they will be able to make representative bulk samples of various materials. Technicians in the Q.C. group will be trained to take precise accurate aliquots from bulk samples, and in the use of the measurement and analysis equipment appropriate to the type of determination being performed.

4.7.3 Documentation

The supervisor responsible for the training of each technician will record the results of each training session, and forward these results to the MCC, who will keep them on file for two years. When the supervisor and the MCC agree that the data generated by the trainee is comparable in quality to data of the same type as provided by qualified personnel, the trainee shall be considered qualified, and only then will measurements which are performed by the trainee be considered acceptable for material control and accounting purposes.