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UNION CARBIDE

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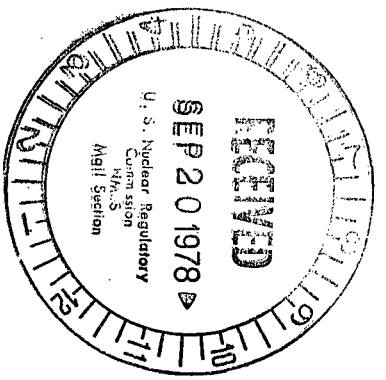


September 18, 1978

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 revised pages of the Union Carbide Corporation Measurement Quality Assurance Program which was last submitted to you for approval on June 23, 1978.

These revisions have been made in accordance with your last set of comments by telephone.

Very truly yours,

James J. McGovern
Manager
Radiochemical Production

JJMcG:js
Enclosures -(pp. 1, 2, 5, 6, 9, 10, 16, & 18 of Chapter 4
(10) to UCC FNMC Plan)

FEE EXEMPT
70.57 update

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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 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 prior to implementation. The Measurement Control Coordinator shall review the entire manual at least annually.

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 Control 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.

4.2.1.1 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 (2 measurements). 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 vertified 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. These standards are made from certified NBS material or equivalent and are verified by analysis (2 measurements).

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.

4.2.1.4 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. Standard Waste Barrels will be representative of the full range of the normal process waste barrels.

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 (at 95% confidence level). 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 for each measurement system being used (based on two standard 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.

4.2.4.1 Bias (cont'd)

1. The estimated bias is less than:

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

   b) 50 grams U-235 total for a material balance period.

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.

4.4.1 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 $\gamma$ 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.

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.