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ACCOUNTABILITY AND SAFEGUARDS PROCEDURES

FOR SNM UNDER

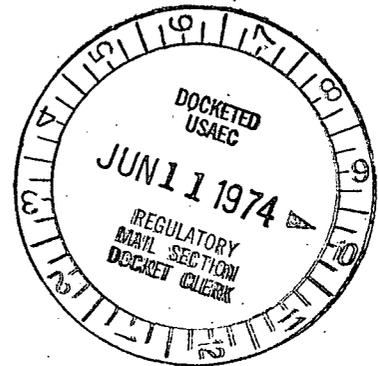
LICENSE SNM-639

UNION CARBIDE CORPORATION

STERLING FOREST RESEARCH CENTER

ORANGE COUNTY - TUXEDO, NEW YORK 10987

May 22, 1974



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1. ORDERING of SNM

- 1a. Only authorized individuals shall initiate an order for SNM. The currently authorized individuals are:

Site Accountability Officer: C. J. Konnerth  
J. J. McGovern (Alternate)

Authorized Individuals: J. L. Ditton  
J. P. Dise  
W. Fecych  
D. D. Grogan  
H. C. Hart  
A. B. Innis  
A. K. Thornton

- 1b. All purchase requisitions shall be endorsed by the Site Accountability Officer.
- 1c. When ordering SNM the U<sup>235</sup> Master Log sheet shall be referred to in order to ensure that the overall license limit will not be exceeded.
- 1d. The Site Accountability Officer will prepare the necessary SNM order form (AEC OR-640) and transfer form (AEC 741) and submit them to the Purchasing Department.

The maximum quantity of SNM that may be possessed on site under License SNM-639 is 2600 grams U<sup>235</sup>.

2. RECEIVING of SNM

- 2a. Only authorized individuals shall open shipping containers containing SNM.
- 2b. All incoming material will be directed to the Health Physics Department, and they will supervise the unpacking procedure.
- 2c. The contents of the shipment shall be checked by weighing against the shipping paper or manifest, and the original purchase request. Any discrepancy among these numbers must be reported to the Accountability Officer. If the quantity received results in exceeding the current license limit, the material must be divided and stored so that our license limit for storage shall not be exceeded and arrangements for returning any excess quantities shall be initiated immediately.
- 2d. The Health Physics Technician will assure that the material is logged into the storage area inventory sheet and the master log sheet, and the Accountability Officer is informed of its arrival.

2. RECEIVING of SNM (Cont'd)

- 2e. All SNM shall be locked in authorized storage cabinets after the quantity of material is verified.
- 2f. All requests for use of  $U^{235}$  from the storage cabinet will be directed to the Health Physics Department who will maintain custody of the storage cabinet keys.
- 2g. Under Health Physics supervision, authorized individuals shall sign material out of the storage cabinet and log it in their inventory sheet for a work area. The Health Physics Technician will assure that the total inventory in the work area does not exceed the process specification limit.

3. STORAGE for SNM

- 3a. Approved storage cabinets shall be provided so that all SNM can be stored in a safe location. (Insurance underwriter approval of storage cabinets is deemed sufficient for this purpose.)
- 3b. The amount of SNM which shall be stored in any one storage compartment, shall be limited to 650 grams.
- 3c. The storage compartment shall be placed so that there is a minimum of 3 feet distance between them and each shall be fixed to prevent inadvertent movement.
- 3d. All requests for withdrawal of  $U^{235}$  from storage shall be directed to the Health Physics Department (Site Accountability Officer), who will maintain custody of the storage cabinet keys. (See Appendix D, "Procedures Pertaining to Locks Safeguarding SNM".)
- 3e. Only authorized individuals may withdraw SNM from storage. All withdrawals will be supervised by a Health Physics Technician.
- 3f. All withdrawal transactions shall be recorded in the appropriate log sheets.

4. PROCESS and/or IRRADIATION of SNM

- 4a. All operations with SNM shall be approved by the Nuclear Safeguards Committee.
- 4b. All operations with SNM shall be performed only in areas authorized under SNM License No. 639.
- 4c. Irradiation of SNM shall be performed as described in the R-81 License.
- 4d. SNM shall not be released to a work area until an authorized individual signs it into the work area and the appropriate log entries are made.
- 4e. SNM in solution shall be limited to a quantity of 300 grams of  $U^{235}$  in a single laboratory.

4. PROCESS and/or IRRADIATION of SNM (Cont'd)

- 4f. SNM for irradiation shall not exceed 650 grams of  $U^{235}$  in a single laboratory.
- 4g. Encapsulated Uranium for irradiation shall be kept in locked storage compartments in an authorized laboratory in a restricted area (laboratory authorized under License SNM-639).
- 4h. SNM in process or in storage in a hot cell shall not exceed 650 grams.
- 4i. Other SNM and/or source material shall not be permitted in hot cells or laboratories containing  $U^{235}$ .
- 4j. Reflection material such as beryllium and deuterium shall not be permitted in hot cells or laboratories containing  $U^{235}$ .
- 4k. A running inventory of SNM in any one location is to be maintained by the authorized individual who is the custodian of the SNM in that location.
- 4l. When unirradiated Uranium is transferred between locations or transformed into a new physical form, an assay shall be performed.

5. INVENTORY of SNM

- 5a. A physical inventory shall be performed every 2 months.
- 5b. Inventories are accomplished through the assay procedures described in the below listed appendix:
  - Appendix A - Assay of Uranium in solution.
  - B - Assay of Uranium in form of  $UO_2$ .
  - C - Assay of Uranium Oxide plated on irradiation targets.
- 5c. A master log sheet containing a running inventory of all SNM possessed under License SNM-639 is maintained by the Site Accountability Officer.
- 5d. A running inventory of SNM shall be maintained by an authorized individual in each laboratory or hot cell where  $U^{235}$  is located.
- 5e. An inventory of SNM shall be conducted upon receipt or shipment of SNM.

5. INVENTORY of SNM (Cont'd)

- 5f. A physical inventory of material in the storage cabinets is performed by actually locating each storage container and noting the current contents of the container log sheet. (If material has not been weighed within 2 months from previous inventory then material must be weighed.)
- 5g. A physical inventory in a plating laboratory is accomplished by assaying all uranium batches in solution and recording the results on an inventory sheet in the log book.
- 5h. A physical inventory of uranium in targets is performed by actually locating each target in storage and noting the quantity of U<sup>235</sup> recorded on the target Q.C. control sheet.
- 5i. A physical inventory of the irradiated uranium stored in a hot cell is performed by locating the waste bottle for each processing batch and noting the quantity of uranium contained in that batch as recorded in the respective target Q.C. control sheet.
- 5j. The MUF (material unaccounted for) shall not exceed an amount allowed by the following LE (limits of error):
  - (a) UO<sub>2</sub> (bulk dry powder)..... ± 0.1%
  - (b) UO<sub>2</sub> encapsulated for irradiation..... ± 3.0%
  - (c) Uranium in solution..... ± 1.5%

After each physical inventory is completed the balance on hand must be reconciled with the balance on record within 30 days after the date of inventory. The record should then be adjusted to show the correct amount found in the physical inventory.

- 5k. All records of SNM transactions and inventories must be held for a minimum of 5 years.
- 5l. Physical inventories of SNM in storage will be conducted by the Site Accountability Officer or his designee. Physical inventories of SNM in process will be conducted by the Supervisor Radiochemical Production or his designee.

6. DISPOSAL

- 6a. Residual uranium, where possible, should be salvaged and recycled into the process chain. When this is done, the appropriate log entries must be made.
- 6b. Waste solutions containing unirradiated uranium shall be contained in vessels which are identified with the contents. These vessels shall be kept in authorized laboratories. The quantity of uranium contained therein shall be noted in the process records of the laboratory where it is stored.
- 6c. Waste solutions containing irradiated uranium shall be contained in vessels marked with the process batch number. These vessels shall be kept in a hot cell.

6. DISPOSAL (Cont'd)

- 6d. All irradiated uranium shall be disposed of in solid form inside a metal container. Shipments will be made in DOT approved containers to licensed burial sites. All unirradiated uranium shall be disposed of in solid form inside a polyethylene container.
- 6e. All disposal operations will be carried out under the supervision of the Superintendent of Nuclear Operations or the Health Physics Supervisor.
- 6f. When SNM is disposed of, a physical inventory shall be performed and the proper log entries shall be made.
- 6g. When SNM is disposed of, a transfer report form (AEC-741) shall be filed by the Site Accountability Officer.

7. PHYSICAL SECURITY

- 7a. The Physical Security Plan for SNM under SNM-639 is enclosed as Appendix "E".

DETERMINATION OF URANIUM IN ELECTROLYTE SOLUTIONSSCOPE:

This method is designed for the determination of total uranium in the electrolyte solutions used in the  $^{235}\text{U}$  target production.

PRINCIPLE OF METHOD:

An aliquot of the solution is fumed with  $\text{H}_2\text{SO}_4$ , the uranium is reduced in the Jones Reductor and titrated with  $\text{KMnO}_4$ .

SPECIAL APPARATUS AND REAGENTS:

Nine-inch Jones Reductor - Place a perforated porcelain plate in the bottom of the reductor tube, followed by a small wad of glass wool. Fill to the neck with amalgamated zinc. Prepare the zinc as follows: shake 800 g. of 20 to 30 mesh zinc with 400 ml. of  $\text{HgCl}_2$  (25 g. per liter) in a liter flask for 2 minutes. Wash several times with  $\text{H}_2\text{SO}_4$  (5 + 95) and then thoroughly with water. Keep the reductor filled with water when not in use.

0.1N POTASSIUM PERMANGANATE:

Stock Solution - Dissolve 6.25 g. of  $\text{KMnO}_4$  in 50 ml. of boiling water. While still hot, filter through glass wool into a 100-ml. volumetric flask and dilute to volume.

0.05N  $\text{KMnO}_4$  - Filter 27.0 ml of stock solution into a 1000-ml. volumetric flask through burned off asbestos and dilute to volume.

Standardization - Weigh 0.3000 g. of sodium oxalate (N.B.S.) into a 600-ml. beaker. Add 250 ml. of  $\text{H}_2\text{SO}_4$  (5 + 95) which has been boiled and cooled to  $27^\circ\text{C}$ . Stir until dissolved and add 39 to 40 ml. of  $\text{KMnO}_4$  solution. Stir slowly and allow to stand until the pink color disappears. Heat to  $55$  to  $60^\circ\text{C}$  and complete titration at this temperature. The end point should remain for 30 seconds. Determine a "blank" using the same volume of  $\text{H}_2\text{SO}_4$  (5 + 95) and subtract. Calculate the normality, adjust to 0.1N  $\text{KMnO}_4$  with water, and restandardize.

(0.300 g. of sodium oxalate (B.S.) is equivalent to 44.78 ml. of 0.1N  $\text{KMnO}_4$  or 22.39 ml. of 0.05N  $\text{KMnO}_4$ .)

PROCEDURE:

Take a 10 ml. aliquot of the electrolyte solution and transfer it to a 400 ml. beaker, add 12 ml. of 1.1  $\text{H}_2\text{SO}_4$ , cover with watch glass and heat till fumes of  $\text{SO}_3$  evolve. Cool the beaker, wash down the sides and watch glass, cover with water and refume to strong  $\text{SO}_3$  fumes.

Adjust the volume of the uranium solution to about 100 ml. and warm on the hot plate. Add  $\text{KMnO}_4$  solution (25 g. per liter) dropwise until a pink color persists. Cool to room temperature.

DETERMINATION OF URANIUM IN ELECTROLYTE SOLUTIONS (Cont'd)PROCEDURE (Cont'd)

Prepare the Jones Reductor by passing 100 ml. of  $H_2SO_4$  (5 + 95) followed by 100 ml of water through it. Discard these solutions. Pass the uranium solution through the reductor into the flask receiver. Wash the reductor with 100 ml. of cold  $H_2SO_4$  (5 + 95). Blow clean air through the reduced solution for 5 minutes. Wash the air purge with water and remove from the flask. Titrate with standardized  $KMnO_4$ .

Correct the volume of titrant for the blank.

CALCULATIONS:

$$\frac{(A - B) \times 0.119 \times N (KMnO_4) \times 1000}{W} = \frac{g/l}{U}$$

$$g/l \text{ U total} \times \%E = g/l \text{ }^{235}\text{U}$$

CODE:

- A = volume of titrant used  
 B = volume of titrant used for blank determination  
 N = normality of  $KMnO_4$  solution (standardized by the analytical lab)  
 W = volume of aliquot taken  
 %E = % enrichment, data supplied by supplier of material.

URANIUM, UO<sub>2</sub> ASSAY

STEPS:

1. Remove Uranium Oxide from storage cabinet.
2. Transfer to plating laboratory.
3. Remove bottle from metal container.
4. Weigh bottle on analytical balance.
5. Record gross weight (Tare wt. + UO<sub>2</sub> wt.).
6. Weigh out on single beam trip scale in ventilated area, the predetermined amount of UO<sub>2</sub>.
7. Reweigh bottle on analytical balance.
8. Record weight — (Tare wt. + UO<sub>2</sub> wt.).
9. Repackage bottle containing UO<sub>2</sub> into metal container and return to the storage cabinet.
10. Record transaction in log sheets.

CALCULATION:

A - B = Net wt. UO<sub>2</sub> Removed

UO<sub>2</sub> → U → <sup>235</sup>U

wt. UO<sub>2</sub> x C x %E = wt. of <sup>235</sup>U

CODE:

A = initial gross weight of UO<sub>2</sub> + Tare of bottle (tape + lid and label)

B = gross weight of UO<sub>2</sub> + Tare of bottle (tape + lid and label) after UO<sub>2</sub> removal.

C = Uranium Oxide → Uranium (conversion factor) (suppliers' assay data)

E = enrichment factor. (suppliers' assay data)

A. URANIUM, UO<sub>2</sub> ELECTROLYTICALLY DEPOSITED ASSAY

STEPS:

1. Weigh target on analytical balance after preplating step and air drying. Record weight.
2. Weigh target on analytical balance after stripping and pyrolysis (gross wt.). Record weight.

CALCULATION:

$$(A) - (B) + (C) \times 0.88149 = \text{Net wt. U gms.}$$

$$\text{wt. U} \times \%E = \text{wt. gms. } ^{235}\text{U}$$

CODE:

A = gross wt. of target - gms.

B = tare wt. of target - gms.

C = preplating wt. of UO<sub>2</sub> (gms) established in the development procedure.

%E = % enrichment (supplied by supplier of material)

NOTE:

0.88149 factor for UO<sub>2</sub> → U theoretical conversion factor.

The electrolytically deposited oxide was established to be UO<sub>2</sub> by X-ray diffraction techniques.

B. RADIOMETRIC ASSAY OF PLATED TARGETS

1. Each plated target is placed on the stand above the NaI detector of the upper counting room analyzer. The stand is specifically designed to center the plated region of the target approximately 30 cm above the crystal.
2. The analyzer is set to make a 5 minute count in the range of 0 to 1 MEV.
3. A photo peak area is calculated for the photon energy of 185 KEV and the Uranium-235 assay is determined by the following formula:

$$\frac{(\text{Total CPM in PPA}) - \left( \frac{\text{CPM}_l + \text{CPM}_r}{2} \times \# \text{ channels} \right)}{2463.8 \frac{\text{CPM}}{\text{gm U}^{235}}} = \text{gms. U}^{235}$$

where:

CPM = counts/min.

PPA = photo-peak area

CPM<sub>l</sub> = counts/min. on left edge of PPA

CPM<sub>r</sub> = counts/min. on right edge of PPA

4. The distribution of loading on the target is obtained by making individual counts thru a shielded colimator on a stand 15 cms above the crystal at 3,6,9,12, and 15 inches measured from top to bottom along the length of the target.
5. The PPA is calculated for each of these counts using the formula in step No. 3 above.
6. The % loading at each interval is then calculated by:

$$\frac{\text{CPM}_i}{\sum_{i=1 \rightarrow 5} \text{CPM}_i} = \% \text{ loaded @ } i^{\text{th}} \text{ position}$$

7. These assay results are reported on the target QC control sheet.

PROCEDURES PERTAINING TO LOCKS SAFEGUARDING SNM

(Regulatory Guide 5.12).

1. Door locks should preferably be combination locks which meet the specific requirements of Regulatory Guide 5.12. They should be dial type locks. Key operated door locks are acceptable, but they should meet the specific requirements of Regulatory Guide 5.12.
2. Padlocks, combination type, should be used on storage cabinets. Key operated padlocks are acceptable, however they should be sufficiently strong to resist forcing.
3. Administration of locks, keys and padlocks:
  - (1) Only authorized individuals should know combinations.
  - (2) Combinations should be changed:
    - (a) when there are personnel changes
    - (b) whenever combination is compromised
    - (c) semi-annually
  - (3) Records of combinations should be kept in locations secured with a combination lock.
  - (4) Only authorized persons shall be issued keys to locks.
  - (5) Keys should be checked in and out daily or when necessary.
  - (6) A log of key issues should be maintained.
  - (7) Keys must be recovered from personnel whose assignments have changed.
  - (8) Locks should be changed when:
    - (a) A lock or key is lost
    - (b) A lock or key has been compromised
  - (9) Record of locks and keys should be kept in locations secured with a padlock.
  - (10) A physical inventory of locks and keys should be conducted semi-annually for protection of facilities and bi-monthly when protecting SNM.
  - (11) All locks not in service should be stored in a location which is secured with a combination lock.
  - (12) One person should be placed in charge of locks and keys. The current custodian is J. L. Ditton.

PHYSICAL SECURITY PLAN1. GENERAL INFORMATION

1.1 Title 10 CFR 73.60 requires that all sites possessing certain minimum quantities of SNM must protect such material from theft or diversion in accordance with certain minimum requirements. Title 10 CFR 70.32.f requires that facilities authorized to possess these minimum quantities must have an approved physical security plan. The quantity of SNM authorized under SNM-639 is less than the minimum quantity specified however, this physical security plan is promulgated to comply with the intent of the regulations regarding SNM safeguards and it is compatible with the physical security plan pertaining to the SNM authorized under the R-81 License which was submitted in accordance with Title 10 CFR 50.34.c.

2. DEFINITIONS

2.1 "Protected Area" means those areas within the Reactor and Hot Laboratory buildings.

2.2 Material access areas mean storage cabinets, chemical processing laboratories, and hot cells intended and equipped for working with SNM.

3. STERLING FOREST RESEARCH CENTER SITE (SFRC) SECURITY

3.1 The SFRC security officer is responsible for the physical protection of the protected areas.

3.2 During times other than normal laboratory working hours, a watchman is assigned to make periodic tours of the entire site, including protected areas, and to respond to intrusion alarms at his watch station.

3.3 Access to any building on site is limited to authorized individuals. Visitors are issued badges and must be accompanied by site employees. A visitor's log is maintained in the administration building.

3.4 During times other than normal laboratory working hours, all buildings are locked and access to any building is controlled by site security personnel. Keys are only issued to authorized individuals and a key issue log is maintained by security personnel.

3.5 Procedures for responding to civil disturbances have been established. Emergency Procedures include instructions for contacting local law enforcement agencies.

4. PHYSICAL PROTECTION OF THE PROTECTED AREA

4.1 The Protected Area physical barriers consist of building walls constructed of reinforced concrete or cement block with access doors of steel. All access doors are monitored with an intrusion alarm system which sounds at the watch station of the site security personnel.

#### 4. PHYSICAL PROTECTION OF THE PROTECTED AREA (Cont'd)

- 4.2 The Protected Area is posted and maintained as a "restricted area" as defined in 10 CFR 20.3.(a).14, and admittance is controlled.
- 4.3 Admittance to the Protected Area during normal working hours is limited to authorized persons by locks on access doors and/or authorized persons manning access doors.
- 4.4 Admittance during times other than normal laboratory working hours is controlled by site security personnel. Intrusion alarms are energized during this period.
- 4.5 All visitors to the Protected Area are recorded in a visitors log and they must be accompanied by the employee visited.
- 4.6 Visitors are not allowed to carry parcels or packages into the Protected Area without them being searched. Normally, such items are left with the receptionist.

#### 5. PHYSICAL SECURITY OF MATERIAL ACCESS AREAS

- 5.1 All material access areas for special nuclear material authorized under SNM-639 will be within the Protected Area described in Section 4 of this plan.
- 5.2 SNM in storage (in the form of Uranium Oxide or Metal) prior to (chemical process or irradiation) use will be kept in locked steel cabinets (insurance underwriter approved) in a critically safe array.
- 5.3 SNM in process shall be kept within laboratories, hot cells, or the reactor which are within the Protected Area described in Section 4 of this plan.
- 5.4 Only personnel authorized to have SNM will be allowed to have possession of material in these material access areas.
- 5.5 Keys to SNM storage cabinets will be in the custody of the Site Accountability Officer or his designee.

#### 6. SYSTEM SURVEILLANCE

- 6.1 The Protected Area Intrusion Alarm shall be tested daily and a comprehensive test of all entrances shall be performed semi-annually.

#### 7. RECORDS

- 7.1 A list of all individuals authorized to enter the Protected Area shall be maintained and updated annually.
- 7.2 A list of all individuals authorized to be custodians of SNM possessed in accordance with SNM-639 shall be maintained and updated annually.

## 7. RECORDS (Cont'd)

- 7.3 A key log shall be maintained by the Site Accountability Officer or his designee which shall contain a record of storage cabinet key issues and returns.
- 7.4 A register of visitors entering the Protected Area shall be maintained which shows names, dates, times of visit, origin, and person being visited.
- 7.5 During times other than normal laboratory working hours a report of security inspection shall be completed once per shift.
- 7.6 A watchman's log shall be maintained which will contain a record of each abnormal occurrence, including intrusion alarm annunciation and corrective action taken.
- 7.7 A comprehensive recording system documenting SNM transfers between material access areas and/or authorized custodians shall be maintained.

## 8. TRANSPORTATION

- 8.1 Ordinarily, any quantity of SNM authorized under SNM-639 which is to be transported will not exceed the limits specified in Section 73.30.
  - (a). If SNM is to be transported in quantities in excess of this limit, the provisions of 10 CFR 73.30 thru 73.36 will be adhered to.