DOCKET NO. 70-820

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UNITED NUCLEAR
CORPORATION

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October 14, 1964

Mr. Lyle E. Johnson
Acting Director
Division of Materials Licensing
U. S. Atomic Energy Commission
Washington 25, D. C.

SUBJECT: Experimental Program at United Nuclear's Fuels Recovery Plant

Dear Mr. Johnson:

We have recently discussed with you the feasibility of performing an experimental program at our Fuels Recovery Plant. This program would provide useful technical data regarding impurities but would not comprise productive operation. We would plan to perform this work prior to resuming production.

Transmitted with this letter are 15 copies of a plan to perform an experimental program directed toward identifying the source of product impurities and correcting the process to assure higher product quality. United Nuclear requests your early approval of this experimental program.

You will note that this experimental program is distinguished from production in that

- No product (UO_2) will be made
- only a portion of the plant will be in operation
- the uranium material utilized in the experimental program will be processed and recycled in a "closed loop" system
- a project type organization has been established to carry out the program

On the other hand, the program will be executed under the additional management controls which have been instituted at the Fuels Recovery Plant. Those management controls will be the same as those which will exist during subsequent production.

2 Cys to Compliance 10/15/64

1 copy to Lic. Safety Branch 10/15/64

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ACKNOWLEDGED

Mr. Lyle E. Johnson
Acting Director
Division of Materials Licensing

October 14, 1964

The program description attached contains greater detail than our license applications have contained or will contain. We have intentionally written this program in a detailed manner similar to that of the physical inventory plan. On future license applications, however, we shall be mindful of your suggestions regarding plans vs. procedures and of not submitting too much detail.

We will look forward to your approval of this program. If any questions arise, please contact me so that we may promptly provide additional information or arrange to meet with AEC representatives for discussion.

Very truly yours,

W L Allison

W. L. Allison
Acting General Manager

WLA:jh

Attachments - 15

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UNITED NUCLEAR CORPORATION

Fuels Recovery Plant

Wood River Junction, Rhode Island

DOCKETED USAEC OCT 15 1964 REGULATORY MAIL SECTION DOCKET CLERK

EXPERIMENTAL PROGRAM FOR RESOLUTION OF IMPURITY PROBLEM

OCTOBER 12, 1964

D. F. Cronin
Approved by D. F. Cronin
Director of Licensing

T. J. Collopy
Approved by T. J. Collopy
Project Manager

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I. INTRODUCTION

Following the March 1964 start-up of the United Nuclear Fuels Recovery Plant, it became apparent that the plant was not producing acceptable uranium oxide product. During that production period some 80 kgs. of UO_2 was produced but chemical analysis by United Nuclear and New Brunswick Laboratory of the AEC, indicated unacceptable levels of certain metallic impurities; e.g., titanium, zirconium and iron.

The accompanying Experimental Program is directed toward reducing the levels of these impurities (for example, titanium from approximately 20ppm to the AEC specified level of 1ppm or less) in material already containing them and toward establishing an improved extraction process which will preclude them in the future. The experimental program is not a production start-up in that the objectives as stated below do not include making any uranium oxide product. Further, only a portion of the plant and equipment will be utilized and the experimental material will be contained in a "closed loop" within which it may be recycled several times. No new raw material will be introduced into any process equipment.

Prior to the physical inventory at United Nuclear conducted during September and October, there were approximately 24 kgs. of total uranium as impure ADU as solid and approximately 58 kgs. of total uranium as impure uranium solutions in various parts of the plant. (This total in-process inventory of 82.3 kgs. excludes raw material not yet issued to process.) Some of this material had already been recycled through the dissolution and extraction systems; other material passed through the extraction system only once. Following the physical inventory, the in-process uranium distribution was 22.5 kgs. as solids and 60.2 kgs. total uranium as solutions. The material to be used in this experimental program will include part of these uranium solutions.

II GENERAL DESCRIPTION OF PROGRAM

II A OBJECTIVES

The objectives of the experimental program are to: -

1. Identify the source of the metallic impurities.

2. Develop a process to preclude pickup of these impurities in future production.

3. Develop a rework procedure to remove the impurities from existing material and upgrade it to a quality level meeting the requirements of the "AEC Specification for Recovery of Uranium from Uranium Scrap by Commercial Processors", dated October 1, 1962 (Revised October 29, 1962).

II B PRE-EXPERIMENTAL EQUIPMENT CLEAN-OUT

Execution of the experimental program described below requires:

1. Approximately 31 kgs. total uranium (29 kgs. U-235) as 30 gm/liter uranyl nitrate solution.
2. A thorough cleaning of all process equipment involved (feed tanks, extraction, scrub and strip columns, evaporator, precipitator, product tanks and associated piping).

The 31 kgs. quantity required is a significant reduction from the 70 kgs. previously discussed; this reduction in quantity was achieved by a further detailed review of the plant and program.

Cleaning out of the equipment prior to the experimental program will include the removal of 13.7 kgs. of total uranium presently stored in feed tanks 1-D-9 A, B, C, D, E, F. This material is not suitable for the experimental program due to its low concentration. To remove this material (~200 gallons) from the system and yet preclude the danger of handling and storing many 11 liter bottles, the contained uranium will be consolidated as a crude ADU precipitate which will then be stored away from the test area. It is proposed that the following steps be taken to accomplish the pre-experimental equipment clean-up:

1. The old organic solvent containing the Stoddard kerosene as diluent (which is now in the pulse columns 1-C-6, 1-C-7, 1-C-8 and the solvent surge system consisting of tank 1-D-5 and associated piping) will be pumped through the organic wash column (1-C-9) containing 1 M Na_2CO_3. The uranium will be removed from the solvent and the U-barren solvent stored. The aqueous Na_2CO_3 solution will be analyzed and stored for later rework.
2. The 200 gallons of dilute uranyl nitrate in the 1-C-9 tanks (A, B, C, D, E and F) will be drained batchwise to the OK liquor wash column (1-C-10). Any solvent which is present will be allowed to separate. The aqueous uranyl nitrate will be removed from the column in one gallon bottles and transferred to precipitators 1-D-19 A, B and C. Any organic solvent which is present will be sampled, analyzed and stored in 11 liter bottles for later rework.
3. Impure ADU will be precipitated using the current SOP but under technical supervision due to the low U content of the solution (~5 gallons at ~17 grams per liter).
4. The impure ADU will be filtered on the stainless steel Buchner funnels and then stored as a wet cake in one gallon bottles in the approved storage area.
5. The impure ADU filtrates will be carefully sampled and analyzed and the discardible filtrates removed to the filtrate tanks for eventual discharge to the lagoon. Any filtrates high in U content will be reworked prior to discard using the SOP for rework of ADU filtrates.

The operations described above are not routine and strict supervision will be provided.

Removal of this 13.7 kgs. of U will leave 31.5 kgs. of concentrated impure uranyl nitrate in the OK liquor tanks (1-D-10 A and B). This material will be adjusted (by dilution to 30 g/liter U and addition of nitric acid and aluminum nitrate) to column feed and used in the Experimental Program. This operation to prepare the experimental feed material includes the following steps:

1. The uranyl nitrate in tanks 1-D-10 A and B will be transferred batchwise to precipitator 1-D-20 A.
2. One gallon bottles, one bottle in motion at a time so as not to exceed the 500 gram limit proposed later (Section II F), will be used to transfer the uranyl nitrate from 1-D-20 A to the stainless steel dissolver (1-J-4).
3. Water, nitric acid and aluminum nitrate will be added to the dissolver (1-J-4) to produce a feed solution containing 30 g/l U, 3N HNO_3 and 0.25 M $Al(NO_3)_3$.
4. The adjusted feed will be transferred to the assay tanks (1-D-34) via the filter (1-F-24 A) where it will be weighed and sampled. Any solids collected in the filter will be sampled and analyzed.
5. The accounted-for feed will then be transferred to the overhead feed tanks 1-D-9 (A, B, C, D, E or F).

Following this feed adjustement step and storage of all other uranium in the plant outside the test program area, all equipment to be used in the test would be cleaned, new TBP-kerosene solvent would be prepared and the test program started as outlined in the following proposal.

II E LIMITATION OF EXPERIMENTAL PROGRAM

The experimental program after the pre-experimental equipment clean-out phase will be limited to operation of the purification system at steady state and the collection of samples at steady state to determine the source of the impurity problem. This means that only the equipment listed in II D will be used for the program. As a result no digestion of solid materials will take place and the stainless steel dissolver will be used only for transfer and adjustment of recycle uranyl nitrate. The precipitators will not be used to prepare ADU but will be used only for transfer of recycle uranyl nitrate to the stainless steel dissolver. The only ADU which will be precipitated will be for analytical purposes in the laboratory on samples which are collected.

There will be no solid residue workup necessary during the experimental program. The only anticipated liquid residue containing uranium which will have to be removed from the system during the experimental program will be "spent" sodium carbonate solution from the organic wash column. This solution immediately on withdrawal from the column in 11 liter bottles will be sampled and analyzed. Proper storage prior to future workup will be determined from the sample analysis.

In summary, the program will thus be limited to the movement of 31 kgs. of uranium and no ADU will be produced during the experimental program. The elapsed time for the program plus the Cleanout phase is estimated as three weeks.

II F MATERIAL QUANTITIES

As described previously, following the pre-experimental equipment clean-out phase, the experimental program per se will utilize approximately 31 kgs. total uranium (~29kgs. U-235). So long as this material is confined in geometrically-safe equipment or equipment made safe by contained nuclear poison, nuclear safety is assured. As an additional control on the handling and movement, both planned and unplanned, of material outside the safe-equipment, UNC has imposed the following specification on the experimental program.

> "No more than 500 grams U-235 shall exist at any time outside the process equipment to be utilized in the experimental program."

The only predictable causes for any U-235 to exist outside the process equipment are:

1. The movement of 4 liter bottles of solution described in II C.3 during which concentrated uranyl nitrate ~70 g/l is transferred from the OK liquor tank (1-D-10 C) to the feed tanks (1-D-9 A, B, C, D, E or F) via 1-D-20A, the stainless steel dissolver (1-J-4) and the assay tank (1-D-34).

2. The removal of spent Na_2CO_3 or TCE~1 gm U/l from columns 1-C-9 and 1-C-10 respectively.

These two categories of transfer operations will never be performed simultaneously. The spent work solutions Item 2 will be stored after analysis in either 11 liter bottles (for > 1 gm U/liter) or in 55 gallon drums filled with Raschig rings (< 1 gm U/l).

III ORGANIZATION for and CONTROL of the TEST PROGRAM

A. Overall Responsibility

The experimental program, as were all other post-accident activities, will be carried out under the cognizance of the "Go-No-Go Committee" composed of the Division Vice-President, Chemical Operations Manager and Quality Control Manager.

B. Organization

The organization for the experimental program is shown on the following page.

The full-time on-site Project Manager will direct the program, interpret experimental data, and determine the need for any test program modificiations. He will provide periodic progress reports to the "Go-No-Go Committee".

The Director of Licensing, assisted by a full-time on-site criticality (nuclear safety) engineer and a full-time health physics technician, will oversee the entire experimental program for conformance to safety and health regulations as established by license and such additional precautions as they may institute before or during the program.

The Process Engineer will be full time on-site observing the operations and auditing conformance to this program and the existing plant operating documents (See III E below for description of documents).

The Plant Superintendent will direct the production personnel in the actual discharge of the program.

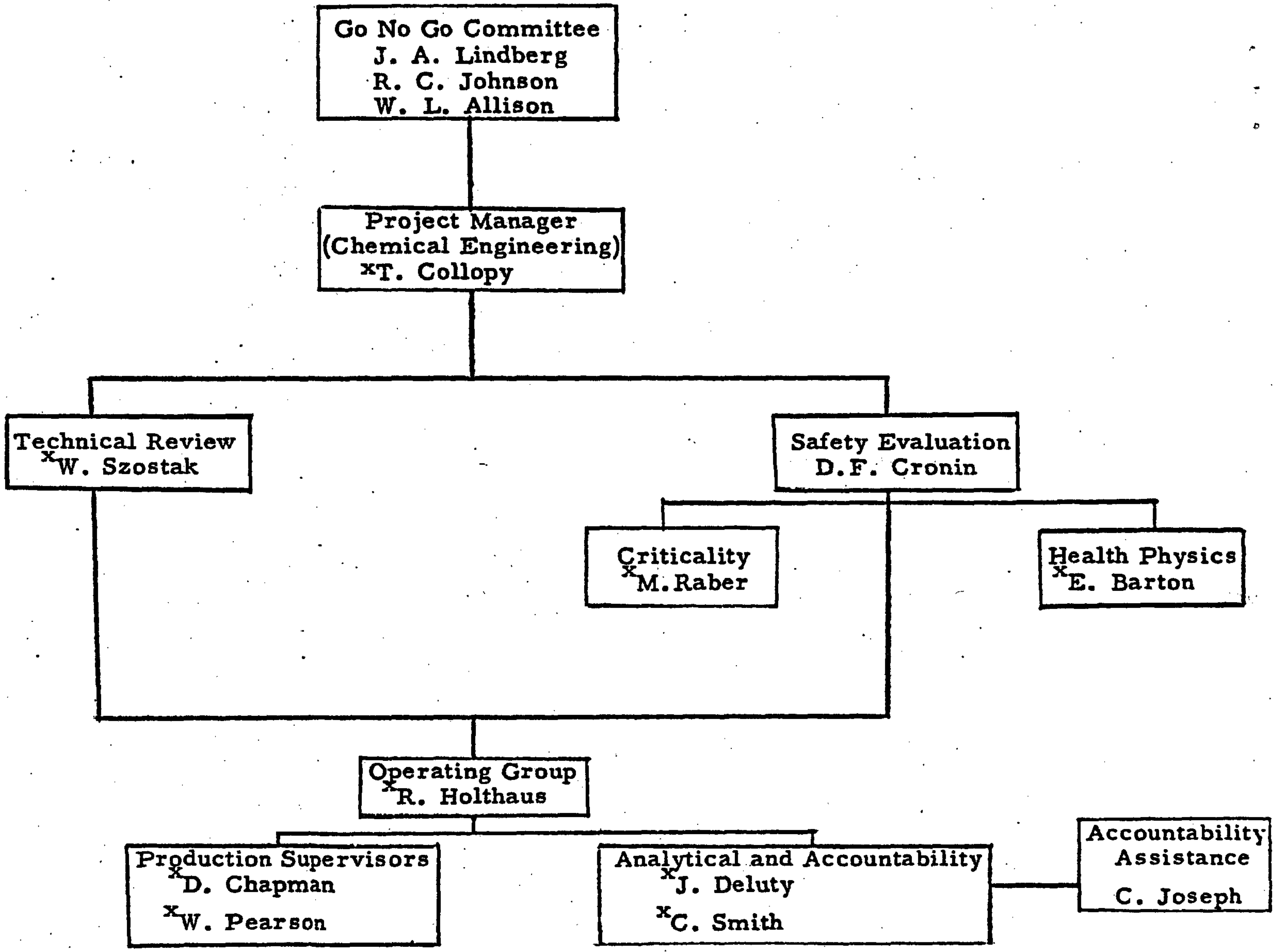
The Plant Chemist will assist in obtaining the analytical samples and perform or have performed the uranium and impurity analyses as requested by the Project Manager and such additional analyses as are needed for accountability or nuclear safety purposes.

C. Program Adjustments

Modifications of the experimental program within the general scope outlined above will be made only after approval by Project Manager with Process Engineering, Nuclear Safety, and Health Physics concurrences, in writing, as program addenda. "General scope" is interpreted to include movement of materials as described in II B and II C above.

No enlargement in program scope will be permitted without approval of the "Go-No-Go Committee" and prior concurrence of AEC.

EXPERIMENTAL PROGRAM ORGANIZATION STRUCTURE



xIndicates on-site, full-time during startup

III ORGANIZATION for and CONTROL of the TEST PROGRAM

D. Personnel Qualifications

Qualifications of key personnel in the experimental program are:

1. Project Manager - T. J. Collopy, currently Group Leader, Chemistry, in Chemical Operations Technical Department, 12 years experience in nuclear fuels processing, 4 years experience in scrap recovery by solvent extraction processes.

2. Director of Licensing - D. F. Cronin, (see license application)

3. Plant Superintendent - R. A. Holthaus, directed plant start-up, 11 years experience in uranium processing and operation of solvent extraction equipment at Goodyear Atomic Corporation.

4. Process Engineer - W. Szostak, Technical Specialist, Fuels Division, six years experience in development in fuels processing.

5. Criticality Engineer - M. Raber, Project Engineer, Criticality Consulting Group, United Nuclear Corporation Development Division. Recently performed independent analysis of Fuels Recovery Plant Nuclear Safety Design and Calculations. Six years experience in reactor physics and nuclear safety.

6. Health Physics Technician - E. Barton, two years experience in this capacity at New Haven and Wood River Junction plants.

7. Plant Chemist - J. Deluty, 17 years experience as analytical chemist, eight months experience in chemistry of uranium.

E. Operating Documents and Controls

All operations at the Fuels Recovery Plant will be performed according to authorized and approved procedures. To effect management control in this operational sector, eight (8) basic operating documents have been established for future use in the plant:

1. Standard Operating Procedure
2. Process Parameter Sheet
3. Operating Reports
4. Check List
5. Rework Material Notice (RMN)
6. Route Card
7. Supervisors Log Book
8. Daily Production Report

III ORGANIZATION for and CONTROL of the TEST PROGRAM

E. Operating Documents and Controls (cont.)

Tabulated on the following pages are the significant details regarding each of these documents including

- -objective
- -authorization and approval requirements
- -administrative handling and record retention requirement for each as a part of the permanent plant operating record.

UNITED NUCLEAR CORPORATION
FUELS RECOVERY PLANT

SUMMARY of OPERATING DOCUMENTS

DOCUMENT	OBJECTIVE	AUTHORIZATION & APPROVAL	HANDLING	COMMENT
1. Standard Operating Procedures (SOP's)	To communicate the technical process from Engineering to Production	Process Engineering issues. Approval/concurrence signatures by Nuclear Safety, Health Physics & Production.	Each operator has own copy. All (20) copies controlled.	No operations other than those in SOP manual permitted. SOP's conform to License. Issuing authority can revise within License scope. Other functions can request revision by means of Engineering Change Request.
2. Process Parameter Sheets	To establish and communicate the detailed parameters to be used for a particular job.	Process Engineering issues. Revision of parameters requires Process Engineer approval.	Three (3) controlled copies. Process Engineer gives two(2) copies to Supervisor; Operator initials to acknowledge receipt of his copy from Supervisor. When new job requires new issue, Engineer retrieves obsolete sheets.	Parameter sheet is an addendum to a SOP.
3. Operating Reports	To demonstrate process conformance to the SOP's and Parameter Sheets by the Operator and the plant equipment.	Operator fills in (designed by Process Engineering)	Operator fills in one report per job, batch, and/or shift. Report is reviewed for completeness and conformance to process by Supervisor who initials. Reports retained by Process Engineer.	Monthly audit and review by Process Engineer.

DOCUMENT	OBJECTIVE	AUTHORIZATION & APPROVAL	HANDLING	COMMENT
4. Check List	To demonstrate steady-state operation of the continuous (versus batch) equipment, i.e., the extraction columns and the evaporator	Operator fills in (designed by Process Engineering)	List comprises items to be checked every two hours during run. Supervisor reviews. List retained by Process Engineering.	Monthly audit and review by Process Engineer.
5. Rework Material Notice (RMN)	To control material which is not in the normal process and to bring abnormal plant operating conditions to attention of Management.	Operator	Each RMN is serially numbered. Supervisor reviews and provides operator rework instructions (See Route Card, Item 6) RMN's are retained by Process Engineer.	Control of material is from the viewpoints both of nuclear safety and product quality.
6. Route Card	To provide proper and adequate rework instructions and control over deviating material in plant.	Process Engineering	Process Engineer provides preprinted Route Cards to Supervisor who issues applicable Route Cards to Operator who performs rework. Applicability determination by the Supervisor in accordance with Rework SOP. Route Card cross indexed and physically attached to Rework Material Notice.	Process Engineer assures compliance of Route Card operations to License. An authorized route card is issued for every RMN (see 5 above).

DOCUMENT	OBJECTIVE	AUTHORIZATION & APPROVAL	HANDLING	COMMENT
7. Supervisors Log Book	To provide written record of general plant status.	Shift Supervisor	Other supervisors initial that they have read, Superintendent reads and initials. Permanently retained.	
8. Daily Production Report	To summarize production status and accomplishments and to call attention to Rework outstanding in order to permit production planing and control.	Shift Supervisor	Superintendent reviews and issues changes in production plan(including rework) as indicated.	

IV NUCLEAR SAFETY PROVISIONS

IV A PROCESS AND EQUIPMENT REVIEW

Reference License SNM 777

The equipment and procedures to be used have been reviewed for the nuclear safety and health physics aspects. Uranium inventory not needed for this experimental program will be placed in storage and a rope barrier used to insure separation.

The impure ADU precipitate prepared as indicated in Section II B to consolidate unneeded uranium inventory, will be stored according to the requirements of the present license SNM 777. This storage area will be further restricted for this test by a rope barrier.

Equipment not being used will be physically disconnected. Any deviations from SOP's necessary within the scope of this test will require written approval from both the Project Manager and the Project Criticality Engineer subject to review and possible veto from the Director of Licensing or the "Go-No Go" committee.

IV B REVIEW OF NEUTRON ABSORBERS
(RASCHIG RINGS AND CADMIUM SHEET)

Raschig rings (type EN-1 or equivalent with a minimum 5% natural Boron content) are used as neutron absorbers to insure safety for equipment not geometrically safe per se, where uranium of sufficient concentration could possibly be introduced.

The following items specifically use Raschig rings:

1. The organic hold tank (1-D-5).
2. The entrainment separator section of the evaporator.
3. The "slop" tanks for the extraction raffinate system (1-D-21 A and B).

During the cleanup preceeding this proposed test the present rings will be sampled and inspected. Should the visual inspection and physical measurements or excessive weight loss indicate significant deterioration (reduction of Boron content to 4% or less) the rings will be replaced.

Cadmium sheet 30 mils or greater has been placed around the 6" I.D. cyclone separator on the evaporator as an additional safety precaution.

IV C URANIUM OUTSIDE OF TEST EQUIPMENT

The amount of uranium exclusive of samples removed from the equipment during the test will be restricted to 500 gms. As mentioned earlier in Section II F, these drawoff points are specifically indicated.

All analytical samples will be restricted to a maximum of 10 gms U content. Transportation of samples to other analytical laboratories will be limited to accumulation of 350 gm U maximum (prior analytical work at site will determine the uranium content to ± 20%).