CRITICALITY
CONTROL
OF
FISSILE
MATERIALS

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NUCLEAR SAFETY CONTROL IN THE CHEMICAL PROCESSING FACILITIES OF THE SAVANNAH RIVER PLANT*

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Abstract

NUCLEAR SAFETY CONTROL IN THE CHEMICAL PROCESSING FACILITIES OF THE SAVANNAH RIVER PLANT. The irradiated fuel processing facilities at the Savannah River Plant handle large amounts of enriched uranium and plutonium. Several methods of nuclear safety control are employed. All processing of enriched uranium is in solutions of safe concentration in large equipment; plutonium is treated similarly through the solvent extraction process. Favorable dimension control is used with plutonium in finishing, metal production, and recovery operations where more-concentrated solutions are handled. Many operational controls are used. Some of these are: (1) flow rate control and acidity monitors on feed streams to the mixer-settlers to prevent concentration through reflux, (2) neutron monitors to detect uranium or plutonium accumulation (3) rapid instrumental methods of determining concentration, such as gamma absorptometry and plutonium X-ray counting. Overall, a large degree of administrative control is employed to insure safe operation. A distinctive feature of the administrative control system is the parallel review by the Production and Plant technical organizations. A separate technical organization, the Savannah River Laboratory, under management independent of the Plant, reviews all changes in operation that are outside the limits of established technical standards. The Atomic Energy Commission also has a review function for certain types of documents.

1. INTRODUCTION

The Savannah River Plant, located near Aiken, South Carolina, is operated by the du Pont Company for the United States Atomic Energy Commission. The Plant has facilities to manufacture reactor and target assemblies, to irradiate the assemblies in one of four operating nuclear reactors, and to process the irradiated elements chemically to recover the fissile materials and certain other nuclides of interest. The Plant also produces the heavy water used to moderate its reactors. On the same site is the Savannah River Laboratory, whose primary role is to provide the technical bases for the various manufacturing operations.

The chemical processing facilities are contained in two Separations Areas. As originally designed, both areas were basically identical and consisted of a concrete-shielded, remotely operated and maintained process building (canyon) and the necessary support facilities. One area still employs the Purex (30% tributyl phosphate) solvent extraction process [1] to separate and decontaminate uranium and plutonium from irradiated uranium fuel assemblies that contain less than 1% $^{235}$U. Auxiliary direct-maintenance

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facilities provide for the conversion of the depleted uranium into the oxide and the plutonium into the metal [2, 3]. The other Separations Area was modified to use a more dilute tributyl phosphate solvent extraction process to reclaim uranium from irradiated enriched-uranium fuel elements. Both areas employ mixer-settler-type continuous contactors [4] for the main separation and decontamination operations. Miscellaneous auxiliary processes are added from time to time to recover special isotopes [5].

Because of large throughput requirements for natural uranium in the original Plant specifications, large process tanks were utilized throughout the separations process. Nuclear safety control was required only for those parts of the operation where plutonium could be separated from uranium either deliberately or accidentally, and for the resulting plutonium-containing streams. Conversion of one Separations Area to handle enriched uranium required extension of nuclear safety control to the uranium streams.

2. NUCLEAR SAFETY IN SEPARATIONS PLANTS

2.1. Basic principles

Chemical processing of irradiated reactor fuel and target elements is a complex operation. To assure nuclear safety, it is necessary to have carefully planned written procedures for the operator to follow. Also, adequate instrumentation and analytical support must be provided to verify that the process is being operated within the prescribed limits. This paper describes the application of these principles to the control of nuclear safety in the chemical processing facilities at the Savannah River Plant. Control of safety in reactor operation at Savannah River, employing these same principles, was discussed at an earlier IAEA conference [6]. A comparison of the safety aspects of fuel reprocessing at several major sites has been made by McBride et al. [7]. Administrative control in the separations processes was discussed earlier by Egan and Mowry [8].

2.2. Types of nuclear safety control

A variety of nuclear safety controls are now in use at the Savannah River Plant. Certain nuclear safety control principles were incorporated in the design of the Plant, and others were introduced through process and equipment modifications. For purposes of discussion, we will consider nuclear safety controls in three categories: basic controls, administrative controls, and operational controls.

Basic nuclear safety controls comprise the fundamentals upon which the control of nuclear safety of a process or portion of a process is based: concentration control, mass control, dimensional control, etc. Administrative controls include the various review and approval steps applied to proposed processes and procedures, and the specific directions for nuclear safety that are written into the detailed procedures. Also classed as administrative controls are the training of personnel and the audit or investigative practices used to verify compliance with nuclear safety requirements and to investigate
their violation. The final category, operational controls, consists of instruments and control units built into or used in close conjunction with the process line to measure and control variables related to nuclear safety. We will consider basic controls and operational controls jointly, since they are closely interrelated.

3. ADMINISTRATIVE CONTROLS

Some degree of administrative control is exercised over nuclear safety in any location where fissile materials are handled. This is necessary initially to ensure adequate consideration of the nuclear hazards in the process and to provide a satisfactory plan to prevent nuclear incidents. Continuing surveillance is then required to guard against unauthorized procedural changes or laxity in compliance with existing controls, and to detect unforeseen hazards. It is important that all changes involving nuclear safety which are outside approved standards receive the same detailed review as the original process proposal.

3.1. Review and approval

A brief look at the organization of the Savannah River Plant (Fig. 1) will aid in understanding the review and approval procedures. The Plant is operated by the Explosives Department of the du Pont Company through its Atomic Energy Division (AED). The Plant proper is the responsibility of the Manufacturing Division, while the Savannah River Laboratory is under the separate management of the Technical Division. Within the Plant organizational structure there are two separate line organizations, the Production Department and the Works Technical Department, which are primarily concerned with the operation of the production facilities. These, together with the Savannah River Laboratory and the Atomic Energy Division management, are involved in the review and approval of new processes and major changes in existing operations.

The Savannah River Laboratory supplies the technical basis and the basic operating conditions for a new operation. These are developed through laboratory experiments and pilot plant tests and through calculations of nuclear safety limits by the Theoretical Physics Section of the Laboratory. Technical Standards, which specify limits for basic variables within which the process must be operated, are distributed for review and approval to the Separations Technology Section and AED management. They are also reviewed by the Separations Department, and information copies are supplied to the Savannah River Operations Office of the USAEC. The organizational flowsheet (Fig. 2) illustrates the review at all levels of the parallel line organization, showing the review routes and approval levels required. The parallel but independent review structure at many levels is felt to be a strong feature of the procedure. All reviewers are expected to consider the safety aspects, including nuclear safety if applicable, along with other facets of the proposed standards.

To operate outside the approved Technical Standards or under conditions deviating significantly from normal operation within the Standards, an
approved Test Authorization is required. This permits investigation of process conditions which may lead to an improved process. A Test Authorization is initiated by Separations Technology personnel after careful study of the possible effects on the process. Technical assistance may be requested from various sources to aid in the evaluation. If nuclear safety is involved,
a nuclear safety specialist is provided within the Separations Technology organization, or aid may be requested from the Savannah River Laboratory. The review and approval route for tests outside of Technical Standard limits is essentially the same as for the Technical Standards themselves, as shown in Fig. 2. If the proposed operation is within Standards, the approvals differ only in that approval of AED management is not required.

3.2. Process operating procedures and nuclear safety control points

Once the Technical Standards have been approved, Separations Technology personnel assigned to the process prepare detailed process operating procedures within the framework of the Standards. Such a procedure is essentially a set of step-by-step directions containing operating values for all pertinent process variables, with spaces for entry of data to provide a log of the progress of the operation. In batchwise operations, a copy of the procedure is prepared for each batch of material, identifying the material and specifying the operating parameters for that batch. At points in the process where extra emphasis on nuclear safety is deemed necessary, "Nuclear Safety Control Point" entries are included in the procedure. These are warning notices, printed in capital letters for emphasis, calling attention to the nuclear safety limits which are in effect for that operation. These control points usually require a supervisor's signature to verify that nuclear safety criteria have been met before the operation may proceed. Similar warning notices are used in detailed equipment operating procedures which are written by Separations Department personnel. Operating procedures require review and approval by Plant management only (Fig. 2).

3.3. Investigation of nuclear safety limit violations

Any unauthorized operation outside of Technical Standard nuclear safety limits requires formal investigation, as does any operating incident with a serious nuclear incident potential even though no limits were violated. As soon as possible following such an incident, the Separations Department Area Superintendent assembles a committee of five people familiar with, but not directly involved in, the operation. Depending upon the location and the seriousness of the incident, other interested parties may be asked to assist the committee. In the investigation the pertinent facts are ascertained, the causes of the incident are established, and recommendations to prevent recurrence are formulated.

3.4. Personnel training

The necessary training of operating personnel in the basic aspects of nuclear safety may properly be considered a part of the administrative control of nuclear safety. All personnel who are assigned to work with a process involving fissile materials are given a basic indoctrination course in the fundamentals of nuclear fission and the various physical means (basic nuclear safety controls) used to prevent a nuclear excursion. Refresher classes are required at regular intervals. Before start-up of a new process,
personnel are briefed on the nuclear safety aspects of the operation. Records are kept indicating the nuclear safety training each operator has received. No operator is permitted to operate equipment where nuclear safety is a factor unless he has had the basic indoctrination course. In addition to scheduled training periods, immediate supervisors give individual instruction during on-the-job training, including nuclear safety when applicable.

3.5. Nuclear safety audits

Another aspect of administrative control is the audit function. Criticality Audit Committees at two different levels regularly audit plant practices and performance. A Separations Area Committee (consisting of a representative from the Separations Department, one from the Separations Technology Section, and one from the Health Physics Section) audits selected process areas four times a year. Operating procedures are examined for adequate nuclear safety emphasis, and personnel training records may be reviewed. Process operators, selected at random, may be questioned concerning their understanding of the nuclear safety aspects of their jobs. Storage and process equipment areas may be examined for adequacy of and compliance with the nuclear safety limits. A report is written by the committee chairman to the Superintendent of the Separations Department summarizing the committee's findings and recommendations. A follow-up report by the supervisor of the audited facility stating actions taken on the recommendations is mandatory.

A Plant Criticality Committee (consisting of the General Superintendent of Production, the General Superintendent of Works Technical, and the Director of the Physics Section of the Savannah River Laboratory) considers the nuclear safety controls proposed for a new operation and may also act as a review authority for investigations of operating incidents involving nuclear safety.

4. BASIC CONTROLS AND OPERATIONAL CONTROLS

Nuclear safety in the chemical processing facilities does not depend on any one basic control principle. Large throughput requirements in the design specifications for the plant ruled out either a completely batch-controlled process or a dimensionally "ever safe" operation. Two types of basic nuclear safety controls do predominate; these are (1) concentration control, which is employed principally with the large vessels in the canyon processes, and (2) favourable-dimension control, used generally in the plutonium finishing and recovery operations where more concentrated solutions are handled. "Favourable dimension" refers to the control of one or more vessel dimensions to give a minimum critical mass in the vessel several times the mass in a normal batch. The vessel is safe for all anticipated conditions but not necessarily for all possible conditions. Multiple errors would be required to cause a nuclear excursion.

Both the basic and the operational nuclear safety controls are best considered in relation to specific processes and operations. In this paper, the two main processes - Purex and enriched uranium - will be described briefly, and specific operations will then be discussed in more detail, with
particular reference to nuclear safety controls. The equipment and the
control problems are quite similar for both processes. For this reason,
nuclear safety controls will be described chiefly in terms of the Purex
process, and controls for enriched uranium processing will be considered
chiefly in terms of differences from Purex. Three other processes or
operations will also be considered: the plutonium finishing process, the
plutonium recovery facility, and the receiving basin for off-site fuels. This
discussion is not intended to be exhaustive, but it will serve to indicate the
kinds of controls in use.

4.1. Brief process description

4.1.1. Purex process. Irradiated aluminium-clad fuel elements of natural
or very slightly enriched uranium are declad with sodium hydroxide solu-
tion and dissolved in nitric acid. The dissolver solution is treated with
gelatin to coagulate silica, centrifuged to remove the silica and other solids,
and fed to the first solvent extraction cycle. Here plutonium and uranium
are separated first from fission products and then from each other in mixer-
settlers, with 30% tributyl phosphate-kerosene as a solvent. Product-
containing streams from the first cycle are fed to the second uranium and
second plutonium cycles, where further decontamination takes place. The
uranium is converted to the oxide while the plutonium is reduced to the metal.
A block flow diagram of the Purex process is given in Fig. 3.

4.1.2. Enriched uranium process. Irradiated aluminium-clad fuel elements
of enriched uranium-aluminium alloy are dissolved in nitric acid. The
dissolver solution is concentrated by evaporation, treated with gelatin to
coagulate silica, and centrifuged to remove silica and other solids. The
solution is fed to the first solvent extraction cycle, where the uranium
and neptunium are first partially decontaminated and then separated from
each other with a 3% tributyl phosphate-kerosene solvent. Both product
streams are sent through second solvent extraction cycles to provide further
decontamination. The uranium, after final decontamination with silica gel
beds, is shipped off-site as a dilute solution. The neptunium is converted to
the oxide. A block flow diagram is presented in Fig. 4.

4.2. Dissolving and feed clarification

4.2.1. Purex. Natural uranium slugs are dissolved in large batches in a
cylindrical dissolver. Slightly enriched uranium slugs are also dissolved
in batches, but an annular dissolver is used. The annular crib is designed
to be nuclearly safe when filled to capacity with uranium slugs containing
up to 1% $^{235}$U. As long as the fissile material remains in solution, there is
no nuclear hazard. A potential hazard would exist if the dissolver were
allowed to become acid-deficient or if sodium hydroxide were accidentally
added to the dissolver solution. The bottom of the cylindrical dissolver is
large enough so that the safe mass per unit area would not be exceeded by
the precipitated plutonium. However, the bowl of the centrifuge used to
remove silica and other solids from the dissolver solution is smaller, and
the safe mass could be exceeded if appreciable precipitation occurred. In addition, the tank receiving the cake may accumulate several such cakes and build up an unsafe quantity of plutonium.

Nuclear safety is controlled in this operation in several ways:

(a) An acid analysis is required on the raw metal solution before it can be transferred to the centrifuge.

(b) When centrifuge cakes are accumulated, a running plutonium balance is kept to ensure that the receiving tank cannot contain an appreciable amount
of plutonium. The accumulated cake is analysed periodically to verify the balance calculation.

(c) The process piping arrangement prevents the accidental addition of any chemical that can precipitate plutonium to any tank between the dissolver and the centrifuge.

(d) Neutron monitors on the centrifuge provide a positive indication of plutonium build-up in the cake.

4.2.2. Enriched uranium. Fuel tubes of enriched uranium-aluminium alloy are dissolved in a cylindrical dissolver equipped with a special insert. The insert contains four thin, slab-shaped, vertical compartments in rows of two, into which flat bundles of fuel tubes are placed. Nuclear safety control in these dissolvers involves a combination of basic controls. The compartment thickness is safe for the flat bundle of tubes and for an appreciable quantity of tube fragments in dilute solution. (Experience has shown that very few fragments are formed.) A stainless-steel-clad plate of boron steel between the rows reduces interaction. The charge size is chosen so that, even if the entire solution were evaporated to the bottom of the lower steam coil (2 feet from the bottom of the tank), the maximum safe uranium concentration would not be exceeded.

Sodium hydroxide is not used in the dissolver or in any part of the process which contains appreciable quantities of uranium. When the canyon system was converted from the Purex to the enriched uranium process, the piping was carefully reviewed, and wherever it would have been possible through simple mistakes to add a precipitant to a tank containing uranium or to transfer uranium to other than normal process vessels, the piping was removed or "nuclear safety blanks" were installed. These are special pipe blanks with a collar that is welded in place so that it cannot be removed by mistake. Special authorization is required for removal of a "nuclear safety blank." These blanks are used extensively in the enriched uranium process system and to a lesser extent in the Purex system.

In the evaporator and its feed tank, the batch size is controlled so that the safe mass per unit area will not be exceeded. Several instrument controls on the evaporator would have to fail before overconcentration could occur.

4.3. Solvent extraction

4.3.1. Purex. The continuous solvent extraction process is a carefully balanced system which causes the partition of several ionic species. Transfer of plutonium between the phases is controlled chiefly by adjustment of valence, transfer of uranium chiefly by adjustment of the nitric acid concentration. The proper operation of such a system depends upon accurately maintaining feed solution compositions and flow rates. Operation of a mixer-settler bank with one of several input-stream variables outside of specifications can lead to the gradual accumulation of plutonium in a particular section of the bank. Because the banks are not designed to be infinitely safe, critical concentrations could be approached in a matter of hours. The continuous mode of operation prevents the use of batch controls.
A number of operational controls are used to ensure the safety of the solvent extraction system:
(a) Recording neutron monitors with alarms are used on banks where plutonium accumulation is possible. One type of installation uses a monitor to measure the quantity of plutonium in the central region of the bank. The other type employs two probes which measure the plutonium concentration on either side of the feed stage. A change in the relative concentrations at these two points indicates an unbalance, and corrective action is initiated.
(b) Recording conductivity cells with alarms are used in all bank input streams where maintenance of acid concentration is essential to prevent plutonium reflux.
(c) Dual flow rate monitoring instrumentation is provided where required on bank input streams. Measuring systems consist of an orifice meter and a rotameter in series, together with their respective recorders and alarms. In some instances the rotameter system actuates a flow control device.

4.3.2. Enriched uranium. Solvent extraction operating problems here are similar to those in Purex. Reflux of enriched uranium in the mixer-settler banks must be prevented. Neutron monitors, conductivity cells, and flow measuring devices are used to warn of abnormal conditions. In addition, recording specific-gravity instruments equipped with alarms confirm the concentration of aluminium nitrate, which acts as a salting agent in certain scrub streams. Colorimeters on a continuously circulated sample stream are used to monitor the uranium concentration in selected sections of several mixer-settler banks. The solvent extraction system is operated at somewhat less than hydraulic capacity to assure that instrument response and personnel reaction times are adequate to permit corrective action before a hazardous amount of uranium has accumulated. Each mixer-settler is equipped with a system for complete and immediate shutdown if the abnormal condition cannot be corrected within a specified period.

4.4. Solvent recovery

Associated with the solvent extraction process are facilities for continuous solvent washing to remove radioactive contaminants and degradation products resulting from radiation exposure. A possibility exists that the solvent from the second plutonium cycle could gradually deposit enough plutonium in the alkaline solvent washer to create a hazard. The plutonium concentration in the solvent normally is extremely low, and any gradual build-up would be detected by routine analyses. Only abnormal operation of the solvent extraction process could produce a rapid build-up, and this occurrence would be detected and corrected at the mixer-settler long before a hazardous condition could be reached in the solvent washer.

4.5. Special sampling

In a few cases where nuclear safety may be largely dependent on a correct product assay in a vessel, duplicate samples are taken, at 15 min intervals, from the agitated solution. The samples are analysed by two
different technicians at two different dilutions. The results must agree within a specified precision or re-sampling is required. This technique guards against inadequate mixing of the solution in the vessel and against errors in the analytical procedure.

4.6. Plutonium finishing

After extensive decontamination in the solvent extraction process, the dilute plutonium solution is transferred to the plutonium finishing facility. Here, mostly in unshielded equipment, the plutonium is concentrated, precipitated as the fluoride, roasted, and reduced to the metal. Before the concentration step, the plutonium is handled on a safe-concentration basis and stored in large tanks. Precipitating agents cannot be accidentally introduced into these vessels.

4.6.1. Concentration. Cation exchange resin columns are used to concentrate the plutonium and to provide further decontamination. The present columns are each made up of two segments which are squat cylinders 7 in. high (including a 5-in. resin bed, metal support plates, and voids) by 10 in. in diameter. These segments represent the favourable-dimension type of basic nuclear safety control; they are too large to be infinitely safe but are small enough to permit a batch size several times the "ever safe" batch. The resin columns initially designed for this service, although smaller in diameter and longer, were based on the same principle and are believed to have been one of the first deliberate applications of this type of nuclear safety control to plant design.

Because of fission product accumulation, the two column segments are in a lead-shielded compartment. The two sections of this compartment are separated by a cadmium-polyethylene neutron shield, and each section is equipped with a neutron monitor and alarm to warn of column overloading. Incidentally, the monitors provide a good indication of the build-up of a plutonium heel on the column.

Downstream from the resin columns, all tanks receiving plutonium solutions are of either favourable or safe dimensions. Most are slab tanks having an internal width of 3 to 3.375 inches. The tanks are in cabinets which not only provide containment of possible radioactive contamination but also provide controlled reflection. Slab tanks are mounted in a common plane to reduce tank interactions to negligible levels. Safe mass limits for individual tanks are generally in the high kilogram or tens of kilograms range, but considerably smaller operating limits are imposed to allow for potential interaction with plutonium in the cabinet sump in the event of major leakage.

Two analytical instruments are used by the production operators in the plutonium finishing facility and in the plutonium recovery facility to reduce the time required between sampling and analytical results. Both instruments are checked against normal laboratory analytical procedures at regular intervals. One, the plutonium X-ray counter, measures low energy X-ray emission from plutonium isotopes. Certain other heavy-element isotopes will also be counted if present. The other instrument, a gamma
absorptometer, measures the total heavy-element content of the solution by the absorption of gamma radiation from an external source.

4.6.2. Precipitation. Plutonium is precipitated as the trifluoride in a two-stage system. Initial precipitation takes place in a small cylindrical tank, from which the slurry overflows into a large slab tank, where it is aged to improve filterability. The newly precipitated plutonium fluoride has a tendency to "plate out" on the polyethylene walls of the vessels, especially in the first stage. Therefore, the system is equipped with a neutron monitor to detect build-up and to provide an indication of the quantity present during the precipitation. Close analytical control of the feed solution is employed to prevent overbatching.

The plutonium fluoride slurry is filtered through a Teflon\(^1\) frit in a shallow cylindrical filter boat, after which it is washed and dried. The filtrate is collected in a slab tank equipped with a neutron monitor to detect frit failure.

4.6.3. Reduction. After drying, the fluoride powder is roasted, then transferred to a ceramic crucible and reduced with calcium to the metal in an induction furnace. The dry powder is handled under the same nuclear safety limits as the metal. Strict limitations are placed on the number of filter boats, roasting pans, loaded crucibles, or plutonium metal pieces (buttons) which may be in any given section of process cabinet. Nuclear safety control points are used frequently in the operating procedures, since nuclear safety depends on control of movable plutonium-containing units. Storage of buttons in process line cabinets is not permitted; they must be removed from the line and stored in nuclearly safe shipping containers (birdcages) or in spaced bins.

4.7. Plutonium recovery facility

This facility provides for the recovery of plutonium from various forms of waste materials. Routine operations include recovery from slag-and-crucible material resulting from the plutonium fluoride reduction process, and recovery from liquid wastes accumulated in laboratory analytical procedures. A major non-routine operation is recovery from plutonium-containing wastes from other sites on a contract basis. Equipment is provided for dissolving the plutonium-containing material and for separating the plutonium by anion exchange.

As in plutonium finishing, the basic nuclear safety control is the use of equipment with favourable dimensions. The dissolvers and all large tanks are slabs, while the anion columns and most of the tanks in the metal recovery system are either favourable-dimension or "ever safe" cylinders. Gamma pulse height analysers are used on the large slab dissolver for non-metallic waste dissolution (and, on a trial basis, on a waste tank, the contents of which are subsequently transferred to a large "non-safe" canyon vessel).

\(^{1}\) du Pont's trademark for its fluorocarbon resins.
Because of volume requirements, three of the slab tanks are actually large annular tanks having an inner diameter of 31 in. These tanks are treated like conventional slab tanks except that allowance is made for neutron interaction across the open centre.

5. RECEIVING BASIN FOR OFF-SITE FUELS

An adjunct to the chemical processing facilities is the receiving basin for off-site fuels (RBOF), where fuel from other sites is received, stored, and prepared for dissolution. The basin handles and stores fuels of many types and compositions covering the entire range of $^{235}\text{U}$ enrichment.

Administrative control is used extensively in this facility. Supervisory confirmation of element identification is required before storage is permitted. An inventory detailing the location of every fuel element by number is maintained. The examination by a nuclear safety specialist of the storage conditions for each fuel is required, and a statement of the safe conditions is made a part of the Technical Standards.

The basic nuclear safety controls employed are batch control and dimension control. Batch control is used in the movement of elements. No more elements can be handled at one time than would be safe if arranged in the worst configuration possible for the intact elements (or smallest intact unit being handled). Dimensional control is used in storage. Fuels are stored vertically in long rows on centre-to-centre spacing of 9 or 12 in. between adjacent rows. Each fuel element is individually restrained in its vertical position. Short fuels may be tiered within a row. All fuel must move into the racks, one element or package at a time, at the approximate storage level and may not be carried above the racks.

REFERENCES


There was no discussion on this paper