Export Control Guide:
Spent Nuclear Fuel Reprocessing
and Preparation of Plutonium Metal

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Foreword

This document was prepared to serve as a guide for export control officials in their interpretation, understanding, and implementation of export laws and controls that relate to the international Trigger List for process components, equipment, and materials used to separate plutonium and uranium from irradiated nuclear fuel elements. Particular emphasis is focused on items that are especially designed or prepared because export controls are required for these by States that are party to the Treaty on the Non-Proliferation of Nuclear Weapons.
Acknowledgments

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Introduction

Nuclear Export Controls:
The Non-Proliferation Treaty

The international Treaty on the Non-Proliferation of Nuclear Weapons, also referred to as the Non-Proliferation Treaty (NPT), states in Article III, paragraph 2(b) that "Each State Party to the Treaty undertakes not to provide . . . equipment or material especially designed or prepared for the processing, use or production of special fissionable material to any non-nuclear-weapon State for peaceful purposes, unless the source or special fissionable material shall be subject to the safeguards required by this Article."

Clarifications to the Treaty:
The Trigger List

In its original form, the Treaty did not specifically elaborate on, or define the meaning of "especially designed or prepared" with reference to equipment or material. Further, the terms "processing, use or production of special fissionable material" were not delineated. As a result, necessary definitions and clarifications of the intent of the Treaty were prepared to facilitate its implementation. These clarifications consisted of a list of especially designed or prepared equipment or material referred to as the Trigger List, which is described in International Atomic Energy Agency (IAEA) information circular INFCIRC/209, dated September 3, 1974, and subsequent addenda. Items that appear on the Trigger List require, or trigger the requirement of, safeguards on their export to non-nuclear-weapon states.

Irradiated Fuel Reprocessing Export Controls

Reprocessing was first mentioned in INFCIRC/209, dated September 3, 1974, as follows:
2.3.1 Plants for the reprocessing of irradiated fuel elements, and equipment especially designed or prepared therefor.

The above statement was clarified in an Annex to INFCIRC/209 as follows:

A "plant for the reprocessing of irradiated fuel elements" includes the equipment and components which normally come in direct contact with and directly control the irradiated fuel and the major nuclear material and fission product processing streams.

Only two items of equipment that met these criteria were identified at that time: irradiated fuel element chopping machines and critically safe tanks.

An extensive review, conducted during the early 1980s, of spent nuclear fuel reprocessing led to a Trigger List clarification, which was published in 1985.

The basis for spent nuclear fuel reprocessing export controls is INFCIRC/209/Mod. 3, Annex, published by the IAEA in August 1985, and subsequently republished in INFCIRC/209/Rev. 1, issued in November 1990, and INFCIRC/254/Rev. 1/Part 1, issued in July 1992. The reprocessing Trigger List is the product of a multinational effort over a period of years. In a series of meetings, a multinational group of reprocessing technology holders developed a preliminary list of equipment, materials, and technology. Additional multilateral committee meetings were held to review and refine the list, and after much discussion, the current list was adopted.
Guide's Objective

This guide was prepared to assist export control officials in the interpretation, understanding, and implementation of export laws and controls relating to the international Trigger List for irradiated nuclear fuel reprocessing equipment, components, and materials. The guide also contains information related to the production of plutonium metal.

Reprocessing and its place in the nuclear fuel cycle are described briefly; the standard procedure to prepare metallic plutonium is discussed; steps used to prepare Trigger List controls are cited; descriptions of controlled items are given; and special materials of construction are noted. This is followed by a comprehensive description of especially designed or prepared equipment, materials, and components of reprocessing and plutonium metal processes and includes photographs and/or pictorial representations. The nomenclature of the Trigger List has been retained in the numbered sections of this document for clarity.
The following actions were taken by the working group of technology holders to prepare the Trigger List:

- developed a comprehensive, preliminary list of equipment, materials, and technology;
- established priorities for items on the developed list based on their uniqueness of function or design features;
- developed simplified descriptions that contain key specifications;
- determined domestic and foreign suppliers;
- evaluated potential impacts on suppliers that could result from establishment of controls on each individual item;
- evaluated potential effectiveness of proposed controls;
- identified and determined alternatives as required;
- refined the list of items by avoiding end-use specifications whenever possible;
- initiated bilateral discussions; and
- initiated multilateral discussions among technology holders and key supplier states.

The NPT Exporters' Committee (Zangger Committee) then adopted a Trigger List of irradiated nuclear fuel reprocessing and plutonium production equipment, components, and materials.
Fig. 1. The reprocessing plant plays a central role in the nuclear power fuel cycle. Otherwise, a nuclear fuel system would provide for long-term storage of spent fuel.
Nuclear fission is a source of heat energy that can take the place of burning fossil fuel in the generation of electricity. The nuclear reactor can be regarded as a source of heat that substitutes for the combustion process in a coal-fired steam plant or gas turbine power plant. As with any more traditional source of heat, a fuel is required; in a nuclear power plant, it is uranium or plutonium. The way in which the two elements are obtained and used and the way in which the resulting residues are handled involve a complex series of processes that collectively is called the nuclear fuel cycle. These processes are:

- mining of uranium-containing ore;

- treating the ore (refining) and converting the separated uranium to a chemical form suitable for subsequent processing or use (enrichment or fuel fabrication);

- if required, increasing the $^{235}\text{U}$ concentration via enrichment;

- converting the natural uranium or enriched product into a suitable material for fabrication into fuel rods;

- using the fuel in a power reactor (typical time is 1–3 years), followed by storing the irradiated fuel for several months;

- reprocessing the irradiated fuel in order to separate and recover the uranium and plutonium from the highly radioactive fission products;

- storing the fission products in liquid form at the reprocessing plant for several years, followed by further processing to produce material in a form suitable for ultimate long-term storage or disposal; and

- processing and recycling the recovered plutonium and uranium into suitable form and materials for subsequent reuse in reactors.
FIG. 2. Diagrammatic representation of a reprocessing plant.

1. Fuel storage pool
2. Feed adjustment pool
3. Feed unloading pool
4. Fuel storage pool
5. Disposer loading cell
6. Decommissioning room
7. Feed adjustment cell
8. Separation cells
9. U-purification cell
10. Pu-purification cell
11. Utility room
12. Control room
13. Truck storage
Fig 3. Diagram of the movement of spent nuclear fuel in a reprocessing plant.
Reprocessing of Irradiated Nuclear Fuel

The objective of reprocessing is to segregate the valuable components, uranium and plutonium, from the highly radioactive fission products, all of which are present in spent fuel following irradiation in a nuclear reactor. The separation involves a series of mechanical and chemical processes that must take place under conditions of elaborate shielding and remote control handling to minimize the exposure of workers to the high radiation levels, and of high security to safeguard the recovered fissile material.

Irradiated nuclear fuel (usually in the form of assemblies) normally is transported to a reprocessing plant from a nuclear reactor in a specially designed container called a shipping cask. The fuel is removed from the cask and stored in a pool to permit further decay of the highly radioactive isotopes. The ends of the assemblies are removed and the remaining fuel rods are sheared (via chopping machines) into 5-cm pieces. These fragments are placed in a vessel (dissolver) containing nitric acid where dissolution occurs and the fuel element cladding (hulls) is separated. The resulting aqueous solution of uranium, plutonium, and the fission products is contacted with an organic solution containing a compound (tri-n-butyl phosphate), which combines with the uranium and plutonium, transferring them to the organic phase and leaving the highly radioactive wastes in the aqueous phase (solvent extraction). The plutonium and uranium are separated from each other through additional chemical steps. This process is known by the acronym Purex (Plutonium-uranium extraction) and is the standard separation process in use today.
Fig. 4. Aerial view of British Nuclear Fuel Service's (BNFL's) Thermal Oxide Reprocessing Plant (THORP) located near Sellafield, England. This is a commercial plant (LWR fuel) with a nominal design capacity of 1200 MT/year.

Fig. 5. Aerial view of the U.S. government's Purex plant near Richland, Washington. This was a weapons production facility for the processing of low-exposure, non-LWR fuel. The government suspended operation in 1974.
Fig. 6. Perspective cutaway of the Purex plant near Richland, Washington.
Reprocessing Facilities

A reprocessing site consists of several structures. The principal building is usually divided into two structural sections—hardened and nonhardened—and two utility categories—radiation and ventilation/contamination. The hardened portion of the building contains the reprocessing cells and is designed to withstand the most severe natural phenomenon without compromising the capability to bring the process and plant to a safe shutdown condition. Other parts of the building contain the offices and shops and, while important for normal functions, are not considered essential and are designed to less rigorous structural requirements. Radiation primarily is addressed by providing adequate shielding (4- to 6-ft-thick, high-density concrete walls), behind which the reprocessing operations are conducted remotely.

The Purex process has been successfully implemented in facilities of all sizes ranging from small, laboratory-sized facilities with an annual throughput of up to several hundred kilograms of irradiated fuel to very large production plants with annual capacities of several thousand metric tons. Most small plants use remote manipulators and hot cells for reprocessing; early, medium-scale plants used long open bays or canyons for reprocessing activities, while present day commercial plants have returned to more compartmented remotely operated hot cells.

Another consideration that may affect the size of a reprocessing plant is the enrichment of the fissile material being handled. Highly enriched uranium and concentrated solutions of plutonium must be processed in smaller equipment to prevent criticality. This operation may use smaller facilities. Commercial plants are designed to process fuel containing less than 5% $^{235}$U.
Fig. 7. Aerial view of the reactor building and chemical pilot plant during construction in August 1943. The plant is located near Oak Ridge, Tennessee. The concrete shielding for the radioactive materials processing cells of the pilot plant can be seen to the left of the reactor building. The openings indicate the locations and sizes of the individual cells.

Fig. 8. Operational testing of the Oak Ridge pilot plant started in December 1943. By the end of January 1944, the plant was processing 0.3 MT/day of irradiated uranium.
Fig. 9. Sophisticated remote operations now in use at commercial reprocessing plants.
Although plutonium from solvent extraction is almost chemically pure, additional decontamination from fission products, uranium, and other impurities is required. Most weapons and commercial reprocessing plants use additional solvent extraction cycles to provide this service, but some small-scale plants use an ion exchange process. In ion exchange, plutonium is selectively adsorbed and then desorbed from a resin, separating the element from the remaining impurities.

The plutonium product from the purification step, present in aqueous solution, is precipitated by the addition of one of several compounds (hydrogen peroxide, oxalic acid, or HF). The precipitate is removed by filtration or centrifugation and dried. If peroxide or oxalic acid is used, the resulting product may be converted to plutonium dioxide (PuO$_2$) by additional heating in the presence of oxygen. Plutonium tetrafluoride (PuF$_4$) may be made by the hydrofluorination of plutonium peroxide or dioxide with HF, while PuF$_3$ may be precipitated directly from aqueous solution with HF.

The acknowledged procedure for producing metallic plutonium involves the reduction of plutonium fluoride (either PuF$_3$ or PuF$_4$) with an active metal in a sealed rigid vessel (called a bomb). Calcium has become the metal of choice for this use although there is still some use of magnesium. The process begins by packing a finely divided, well-blended mixture of calcium and PuF$_3$ or PuF$_4$ into a cold-rolled steel reduction bomb containing a liner of dense calcium fluoride that protects the interior walls of the vessel from corrosion. The vessel is sealed, evacuated to remove air, and then backfilled with argon to prevent in-leakage. External heat is supplied by placing the bomb in a resistance furnace, adding heat slowly until the entire mass is at the initial reaction temperature (300°C), and then heating the system rapidly to the actual reduction temperature of 600°C. The reaction takes only a few seconds, but considerable time is needed for the preheating and the postcooling steps. After cooling, the vessel is opened and the liner removed and crushed to free the contents. The metal product is freed from the slag, washed in concentrated nitric acid to remove residue, washed with water, dried in an oven, and remelted in an arc furnace.
Fig. 10. Flow sheet for the production of plutonium metal from plutonium nitrate.

Fig. 11. Aerial view of the plutonium finishing plant near Richland, Washington.
Reprocessing of irradiated nuclear fuel relies on the use of nitric acid to dissolve its major components—uranium, plutonium, and fission products—and to separate them from the cladding (hulls). The solution so obtained is very corrosive and all process surfaces that come into direct contact with this liquid must be fabricated from, or lined with, suitable materials. These materials include low-carbon stainless steels, titanium, zirconium, or other high-quality materials. Holding and storage vessels are usually designed for remote operation and maintenance and may incorporate neutron-absorbing substances (e.g., boron or gadolinium) that will provide control of criticality. Larger equipment sizes are acceptable for process operations that involve solutions of lower concentrations of fissile material, such as the extracting-scrubbing contactors that separate the fission products from low-enrichment uranium fuel, but materials resistant to nitric acid corrosion must still be used.
Description of Controlled Items

This section provides brief descriptions, photographs, and sketches of (1) spent nuclear fuel reprocessing components and equipment and, where needed, a mention of materials of construction that are representative of those found in reprocessing plants using the Purex process; and (2) components and equipment used to produce plutonium metal. Although these facilities have process functions similar to each other, the specific type and configuration of the equipment performing these functions may differ between individual facilities for several reasons: the type and quantity of irradiated nuclear fuel to be reprocessed, the intended disposition of the recovered materials, and the safety and maintenance philosophy incorporated into the design of the facility. Space limitations preclude the inclusion in this guide of all possible variants; however, illustrations representative of France, Japan, the United States, and the United Kingdom are shown as well as some innovative solutions to the problems encountered in reprocessing. Information provided for each item on the control list includes a description of the item, its special design features, and a photograph or sketch of the item. The descriptions are taken verbatim from the Trigger List (INFCIRC/209/Rev. 1 and INFCIRC/254/Rev. 1/Part 1) and appear in italics on the pages that follow.
1. Irradiated Fuel Element Chopping Machines

Description

Remotely operated equipment especially designed or prepared for use in a reprocessing plant as identified above and intended to cut, chop or shear irradiated nuclear fuel assemblies, bundles or rods.

Introductory Note

This equipment breaches the cladding of the fuel to expose the irradiated nuclear material to dissolution. Especially designed metal cutting shears are the most commonly employed, although advanced equipment, such as lasers, may be used.

Explanatory Comments

In addition to fissionable and fertile materials, solid fuel elements contain nonfissionable materials such as cladding, bonding agents, mechanical supports, spacers, coolant-flow directors, moderators, and fissionable-atom diluents for decreasing the heat flux. By removing these materials before the dissolution step, valuable chemicals are saved and the quantity of wastes reduced.

Mechanical removal of these extraneous inert materials has been the method of choice in most reprocessing plants. Early systems used blade sawing, abrasive sawing, and cladding removal by extrusion, rolling, milling, or hydraulic expansion.

Currently, fuel elements/rods are chopped into small pieces using a shear. The fuel element or rods are supported on an anvil and a hydraulically operated blade is lowered with force sufficient to shear off short sections of the fuel. Prototype laser systems are now being tested for commercial application.

The major attribute of fuel element chopping machines is their need to be operated and serviced remotely. A desirable feature is prolonged, continuous service under hostile conditions.
Fig. 12. Modular design of a shear pack permits rapid interchange of units for refurbishment in a purpose-designed remote maintenance area. The system has been developed for BNFL's THORP.

Fig. 13. Full-scale experimental shearing machine being developed by BNFL.
Fig. 14. Diagrammatic representation of the fuel element decanning and shearing machine used at the Windscale plant in the United Kingdom.

Fig. 15. Fuel element decanning and shearing machine used at the Windscale plant in the United Kingdom. Equipment is designed to process magnesium alloy-clad fuel (Magnox).
Fig. 16. Power Reactor and Nuclear Fuel Development Corporation's (PNC's) fuel element chopping machine.

Fig. 17. Prototype of PNC's laser fuel element cutting machine.
2. Dissolvers

Description

Critically safe tanks (e.g. small diameter, annular or slab tanks) especially designed or prepared for use in a reprocessing plant as identified above, intended for dissolution of irradiated nuclear fuel and which are capable of withstanding hot, highly corrosive liquid, and which can be remotely loaded and maintained.

Introductory Note

Dissolvers normally receive the chopped-up spent fuel. In these critically safe vessels, the irradiated nuclear material is dissolved in nitric acid and the remaining hulls removed from the process stream.

Explanatory Comments

Major considerations in dissolver design are to provide a container that is corrosion resistant, critically safe, easily loaded with fuel and dissolvent, and readily accessible for product, cladding, and gaseous waste removal. The vessel must also be designed for remote maintenance.

Dissolvers have been classified under two headings. The most common has to do with fuel addition and the method of operation. Hence, dissolvers are usually referred to as batch or continuous. A second common designation has to do with the shape of the vessel (to prevent criticality) and leads to the names column, slab, and pot.
Fig. 18. PNC's spent fuel distributor in the dissolver cell.

Fig. 19. Diagram of a distributor and a recirculating dissolver.

Fig. 20. PNC's recirculating dissolver.
Fig. 21. The continuous rotary dissolver (wheel configuration) in the shearing and dissolution facility at La Hague, France.
Fig. 22. Continuous rotary dissolver (horizontal screw type) developed in the United States. It has a capacity of 240 kg of heavy metal per day. The sheared fuel drops into the dissolver through the piping located near the top center of the photograph. Fresh concentrated nitric acid enters the dissolver through piping located at the lower left of the photograph.
Fig. 23. The stainless steel screw system in the horizontal rotary dissolver rocks the sheared fuel for ~30 minutes and then moves forward one rotation. Total residence time is ~4 hours.

Fig. 24. The horizontal rotary dissolver is surrounded by a steam jacket. The material lying in the jacket is discarded cladding from a cold test run.
3. Solvent Extractors and Solvent Extraction Equipment

Description

Especially designed or prepared solvent extractors such as packed or pulse columns, mixer settlers or centrifugal contactors for use in a plant for the reprocessing of irradiated fuel. Solvent extractors must be resistant to the corrosive effect of nitric acid. Solvent extractors are normally fabricated to extremely high standards (including special welding and inspection and quality assurance and quality control techniques) out of low carbon stainless steels, titanium, zirconium, or other high quality materials.

Introductory Note

Solvent extractors both receive the solution of irradiated fuel from the dissolvers and the organic solution which separates the uranium, plutonium, and fission products. Solvent extraction equipment is normally designed to meet strict operating parameters, such as long operating lifetimes with no maintenance requirements or adaptability to easy replacement, simplicity of operation and control, and flexibility for variations in process conditions.

Explanatory Comments

For nuclear application, extractors (also known as contactors) have been designed to be as small as possible, to be resistant to nitric acid, to mix and separate the two solutions as quickly as possible, and to prevent criticality. The major type of contactors used in the nuclear industry include mixer settlers, columns, and centrifugal contactors.

Mixer settlers are relatively simple, have good hydraulic properties in terms of stability, and
permit mixture pass-through under stirrer failure. The stirrer drives can be remotely located from the radioactive solutions so that no radiation or contamination problem exists during their replacement or maintenance. A quiescent volume must be provided to allow the liquids to separate.

Packed columns have given way to pulsed columns in which the liquid contents are pulsed through a column bridged by perforated plates. This change has reduced column heights by 50%.

Centrifugal contactors combine the advantages of short residence time (less exposure of the organic extractant to the highly radioactive aqueous phase) and stage-wise extraction in a small piece of equipment. Centrifugal force is utilized to achieve rapid phase separation after the liquids are mixed, thus eliminating the large settling volume and reducing the contact time of the mixer settler.
Fig. 25. Diagrammatic representation of a three-stage mixer settler.

Fig. 26. Model of a small mixer settler.

Fig. 27. Diagrammatic representation of a first-cycle extraction pulsed column.
Fig. 28. Full-scale pulsed column test system at a reprocessing facility in the United Kingdom.
Fig. 29. A pulsing pump used in the first reprocessing pilot plant in the United States. Capacity of the facility was 0.3 MT of fuel per day.
Fig. 30. A cutaway of a pulse column showing the perforated plates that mix the organic and aqueous phases during the pulsing period.
Fig. 31. Schematic diagram of a centrifugal contactor.
Fig. 32. A bank of pilot-scale centrifugal contactors.
Fig. 33. The components of a pilot-scale centrifugal contactor.

Fig. 34. Set of centrifugal contactors ready for use in a weapons production reprocessing plant at the DOE Savannah River plant near Aiken, South Carolina.
4. Chemical Holding or Storage Vessels

Description

Especially designed or prepared holding or storage vessels for use in a plant for the reprocessing of irradiated fuel. The holding or storage vessels must be resistant to the corrosive effect of nitric acid. The holding or storage vessels are normally fabricated of materials such as low carbon stainless steels, titanium or zirconium, or other high quality materials. Holding or storage vessels may be designed for remote operation and maintenance and may have the following features for control of nuclear criticality:

(1) walls or internal structures with boron equivalent of at least two per cent, or

(2) a maximum diameter of 175 mm (7 in) for cylindrical vessels, or

(3) a maximum width of 75 mm (3 in) for either a slab or annular vessel.

Introductory Note

Three main process liquor streams result from the solvent extraction step. Holding or storage vessels are used in the further processing of all three streams as follows:

(a) The pure uranium nitrate solution is concentrated by evaporation and passed to a denitration process where it is converted to uranium oxide. The oxide is reused in the nuclear fuel cycle.
(b) The intensely radioactive fission products solution is normally concentrated by evaporation and stored as a liquor concentrate. This concentrate may be subsequently evaporated and converted to a form suitable for storage or disposal.

(c) The pure plutonium nitrate solution is concentrated and stored pending its transfer to further process steps. In particular, holding or storage vessels for plutonium solutions are designed to avoid criticality problems resulting from changes in concentration and form of this stream.

Explanatory Comments

Criticality may be controlled by limiting the mass of fissile material, its concentration, or its configuration. The restriction of one dimension (slab) or two dimensions (cylinder) to less than the minimum critical value permits the accumulation of much more aqueous solution than can be allowed by mass limits. An operational problem appears when solutions leave the limited geometry of a storage vessel. Precautions must be taken to ensure against the carryover of sludges, fines, and precipitates of fissionable material and to ensure that the receiving vessel is critically safe.
Fig. 35. Storage vessels used for uranium purification, concentration, and denitrification.

Fig. 36. Vessels used for plutonium purification and concentration.
Fig. 37. Vertical configuration of tanks for the storage of concentrated solutions of fissile material. These vessels were used in the first reprocessing pilot plant in the United States.
Fig. 38. Specially designed tanks for storing solutions containing high concentrations of fissionable material. Note spacing of the vessels to prevent criticality.
5. **Plutonium Nitrate to Oxide Conversion System**

**Description**

Complete systems especially designed or prepared for the conversion of plutonium nitrate to plutonium oxide, in particular adapted so as to avoid criticality and radiation effects and to minimize toxicity hazards.

**Introductory Note**

In most reprocessing facilities, this final process involves the conversion of the plutonium nitrate solution to plutonium dioxide. The main functions involved in this process are: process feed storage and adjustment, precipitation and solid/liquor separation, calcination, product handling, ventilation, waste management, and process control.

**Explanatory Comments**

This system may be identified by the measures taken to avoid criticality (e.g., by geometry), radiation exposure (e.g., by shielding), and toxicity hazards (e.g., by containment).
Fig. 39. Ion exchange resins in critically safe pipes are used to provide additional decontamination from uranium and fission products before the plutonium nitrate is converted to the oxide.
6. Plutonium Oxide to Metal Production System

Description

Complete systems especially designed or prepared for the production of plutonium metal, in particular adapted so as to avoid criticality and radiation effects and to minimize toxicity hazards.

Introductory Note

This process, which could be related to a reprocessing facility, involves the fluorination of plutonium dioxide, normally with highly corrosive hydrogen fluoride, to produce plutonium fluoride which is subsequently reduced using high purity calcium metal to produce metallic plutonium and a calcium fluoride slag. The main functions involved in this process are: fluorination (e.g. involving equipment fabricated or lined with a precious metal), metal reduction (e.g. employing ceramic crucibles), slag recovery, product handling, ventilation, waste management and process control.

Explanatory Comments

This system may be identified by the measures taken to avoid criticality (e.g., by geometry), radiation exposure (e.g., by shielding), and toxicity hazards (e.g., by containment).
Fig. 40. Plutonium metal is produced by reducing plutonium fluoride with an active metal, usually calcium. A mixture of the two compounds is placed in a cylindrical chemical reaction vessel (called a "bomb" because of the powerful reaction) containing a liner of CaF$_2$ and the vessel is sealed, placed in a furnace, and heated until the reduction occurs.
Fig. 41. Plutonium metal is removed from the chemical reaction "bomb" and separated from the slag.
Fig. 42. A technician at the U.S. Rocky Flats weapons facility inspects a figure-eight ingot of plutonium.