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Supersedes RDT E 13-6T

June 1971

nuclear STANDARD

**FAST BREEDER REACTOR
MIXED OXIDE FUEL PELLET**

JANUARY 1984

*U. S. DEPARTMENT OF ENERGY
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FOREWORD

This standard supersedes the June 1971 issue of RDT E 13-6T and incorporates those changes to that issue of the standard that were approved and published as Amendment 1 and those changes that were approved for publication in this revision. Because of extensive changes, the location of changes are not identified.

Significant changes that have been incorporated in this revision are summarized as follows:

1. Incorporation of FFTF fuel pellet procurement experience.
2. Revisions of text to be more concise and more generally applicable by referencing the Ordering Data for certain technical specifications.
3. Deletion of certain quality assurance requirements that are adequately covered by reference to ANSI/ASME NQA-1, and NE F 3-15T.
4. Updating of referenced standards.
5. Addition of Systeme International (SI) metric units.
6. Format changes in accordance with the current requirements for DOE Nuclear Energy Program Standards.

Editorial changes have been made to conform to the Department of Energy nuclear energy program decision to designate the RDT standards as NE standards rather than as NE(RDT). Some of the referenced standards are in the process of being updated and issued as NE standards. To minimize editorial corrections to this standard to correct reference to RDT standards to NE standards as they are issued, the corrections are being made prior to issuance of the RDT standards as NE. These changes are not identified.

FAST BREEDER REACTOR MIXED OXIDE FUEL PELLET

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FAST BREEDER REACTOR MIXED OXIDE FUEL PELLET

1. SCOPE

This standard establishes the requirements for mixed uranium-plutonium oxide fuel pellets to be used in FBR fuel pins.

1.1 Classification. This standard describes the basic requirements for FBR fuel pellets. The purchaser will designate and specify the type(s) of fuel (e.g., enrichment) in the Ordering Data.

1.2 Definitions.

1.2.1 FBR. Fast Breeder Reactor.

1.2.2 Lot. Pellets produced from one batch of blended mixed-oxide powder, consecutively pressed and sintered in one furnace batch or continuous furnace run and submitted for inspection. For sampling acceptance of specific pellet characteristics, a lot may be defined as a population of pellets which would be expected to be homogeneous for the characteristic in question.

1.2.3 LTPD. Lot tolerance percent defective, as defined in NE F 2-7.

1.2.4 β Risk. Purchaser risk as defined in NE F 2-7.

2. APPLICABLE DOCUMENTS

The following documents are a part of this standard to the extent specified in Sections 3 through 5. The issue of a document in effect on the date of invitation to bid, including any amendments or other published changes also in effect, shall be as specified in the Ordering Data. Where this standard appears to conflict with the requirements of a referenced document, such conflict shall be brought to the attention of the purchaser for resolution.

2.1 NE Standards.

NE E 13-1T Ceramic Grade Plutonium Dioxide

NE E 13-2T Ceramic Grade Uranium Dioxide

NE E 13-5T Fast Breeder Reactor Fuel Pin

NE F 2-6T Qualification and Control of Analytical Laboratories for Mixed Oxide Fuel Analysis

NE F 2-7T	Acceptance Sampling Plans
NE F 3-10T	Nondestructive Examination of Reactor Core Components and Test Assemblies
NE F 3-15T	Requirements for Inspection of Dimensional Characteristics
NE F 11-1T	Analytical Chemistry Methods for Mixed Oxide Fuel
NE F 11-4T	Determination of a Figure of Merit for $\text{PuO}_2\text{-UO}_2$ Fuel Pellet Homogeneity by Use of an Electron Microprobe
NE F 11-5T	Determination of Fuel Pellet Homogeneity by Alpha-Autoradiography
NE F 11-6T	Ceramographic Preparation of Mixed Oxide Fuel Pellets

2.2 American National Standards (ANSI).

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facilities

2.3 American Society for Testing and Materials (ASTM) Publications.

ASTM E 29 Indicating Which Places of Figures are to be Considered Significant in Specified Limiting Values

2.4 U. S. Government Standards and Specifications.

Code of Federal Regulations, Title 49, Transportation, Part 173

2.5 Other Publications. Other publications as specified in the Ordering Data.

2.6 Drawings. Drawings to be as specified in the Ordering Data.

3. TECHNICAL REQUIREMENTS

3.1 Feed Materials.

3.1.1 Plutonium. The plutonium dioxide powder shall meet the requirements of NE E 13-1.

3.1.2 Uranium. The uranium dioxide powder shall meet the requirements of NE E 13-2.

3.2 Fuel Pellet Composition.

3.2.1 Fuel Pellet Fissile Concentration. The fuel pellet fissile concentration (percentage $^{239}\text{Pu} + ^{241}\text{Pu}$ in pellet) shall meet the requirements for the pellet type required on drawings specified in the

Ordering Data. The fissile concentration shall be reported as a percentage of $^{239}\text{Pu} + ^{241}\text{Pu}$ in a pellet. Conformance with the fuel pellet fissile concentration requirements shall be established by use of formula and calculations in 6.3. The probability of accepting any given inspection lot of pellets having a LTPD as high as 5, shall not exceed a β of 0.05.

3.2.2 Fuel Pellet Plutonium.

a. Concentration. Fuel pellet plutonium concentration (percentage of plutonium in pellet) shall be consistent with the fissile concentration requirement of 3.2.1. The purchaser will supply the reference nominal plutonium concentration for the pellet type required on drawings specified in the Ordering Data.

b. Isotopic Concentration. Fuel pellet plutonium isotopic concentration (percentage of isotopes in plutonium) shall be consistent with the fissile concentration requirement of 3.2.1.

3.2.3 Uranium Concentration. The average uranium concentration for each pellet lot shall be obtained by assay and shall be reported to the purchaser. The 95% confidence interval on the average uranium concentration shall not be greater than $\pm 1.0\%$ of the true mean value.

3.2.4 Impurity Levels.

a. Individual Impurities. Impurity levels shall not exceed the maximum limits shown in Table 1 unless otherwise specified in the Ordering Data. The probability of accepting any given inspection lot of pellets having a LTPD as high as 25 for impurity groups 1 through 11 identified in Table 1 shall not exceed a β of 0.10 unless otherwise specified in the Ordering Data.

b. Total Impurities. Total impurities listed in Table 1 and exclusive of americium, shall not exceed 4000 $\mu\text{g/g}$ by weight unless otherwise specified in the Ordering Data. The probability of accepting all the pellets of the type ordered having a LTPD as high as 10 shall not exceed a β of 0.10 for each impurity group unless specified otherwise in the Ordering Data. The average total impurities for an order, or type of fuel shall be as specified in the Ordering Data.

3.2.5 Americium Concentration. The concentrations of ^{241}Am and ^{241}Pu and the date of analysis for each fuel pellet lot shall be reported as specified in the Ordering Data.

3.3 Oxygen-to-Metal Ratio. The oxygen-to-heavy-metal ratio in individual fuel pellets shall be 1.93:1 to 1.98:1. The probability of accepting any given inspection lot of pellets having a LTPD as high as 10 for oxygen-to-metal ratio shall not exceed a β of 0.10. The lot average of the oxygen-to-heavy-metal ratio shall be such that the fuel column oxygen-to-metal ratio requirement in NE E 13-5 can be met.

Table 1. Fuel Pellet Impurity Limits

Impurity Group	Element	Maximum Impurity Limit $\mu\text{g/g}$
1	Na K	SUM = 700 (No one > 500)
2	Cl F	SUM = 30
3	C N	SUM = 350 (No one > 200)
4	Sm Eu Gd Dy	SUM = 200
5	Fe Cr Ni	SUM = 1500 (No one > 750)
6	P S	SUM = 400 (No one > 300)
7	Zn Pb Sn Cd	SUM = 220 (No one > 100)
8	Be Ca Mg Al Si	SUM = 1000 (No one > 500)
9	B	20
10	Li	10
11	Co Ti V Ta W Cu Ag Mo Mn	SUM = 1000 (No one > 500)
	Zr	700

3.4 Volatiles.

3.4.1 Gas Content. The maximum fuel pellet gas content, exclusive of water, shall be $0.09 \text{ cm}^3 \text{ (STP)/g fuel}$. The probability of accepting any given inspection lot of pellets having a LTPD as high as 10 for gas content shall not exceed a β of 0.10 for the maximum fuel pellet gas content exclusive of water. Exclusive of hydrogen and water, the lot average of gas content shall be such that the requirements of NE E 13-5 can be met.

3.4.2 Moisture Content. The moisture content of individual pellets shall not exceed $20 \text{ } \mu\text{g/g fuel}$. The probability of accepting any given inspection lot of pellets having a LTPD as high as 10 shall not exceed a β of 0.10. The lot average of moisture content shall be such that the requirements of NE E 13-5 can be met.

3.5 Homogeneity.

3.5.1 Homogeneity Figure of Merit. The minimum figure of merit value (M) for fuel pellet plutonium homogeneity shall be 0.96. (See NE F 11-4.) Sample analyses shall be performed in accordance with requirements shown in Table 2, or alternatively, the process may be qualified in accordance with 3.10.1 to meet this requirement.

3.5.2 Plutonium Concentration Regions. The average plutonium concentration of any 300 micron ($300 \text{ } \mu\text{m}$) diameter zone shall not exceed the nominal pellet plutonium concentration for the pellet type specified in 3.2.2 by more than 40% relative; i.e., 1.40 N%. The plutonium concentration of any plutonium concentration zone within any 300 micron ($300 \text{ } \mu\text{m}$) diameter zone shall be no greater than that shown in Fig. 1. Sample analyses shall be performed in accordance with requirements shown in Table 2, or alternatively, the process may be qualified in accordance with 3.10.1 to meet this requirement.

3.6 Grain Size, Porosity, and Secondary Phases. Photomicrographs showing grain size and porosity for each pellet lot inspected for homogeneity figure of merit (3.5.1) and plutonium concentration regions (3.5.2) shall be submitted to the purchaser. There shall be no more than 1.0 volume percent of any secondary phase in any fuel pellet cross section. Sample analyses shall be performed in accordance with requirements shown in Table 2, or alternatively, the process may be qualified in accordance with 3.10.1 to meet this requirement. The photomicrographs shall be taken on the same samples submitted for alpha-autoradiography.

3.7 Fuel Pellet Weight per Unit Length. The fuel pellet weight per unit length (W/L) shall be as specified in the Ordering Data. The probability of accepting any given inspection lot having a LTPD as high as 5 for W/L shall not exceed a β of 0.05. The purchaser will furnish the nominal fuel pellet density as a percent of theoretical density in the Ordering Data.

Fig. 1. Maximum Allowable Plutonium Content vs. Concentration Zone Diameter.

3.8 Dimensions. Fuel pellets shall conform to the dimensional requirements specified in the Ordering Data.

3.9 Surface Condition.

3.9.1 Pits, Chips, or Voids. Fuel pellets having pits, chips and voids shall meet the W/L requirements specified for 3.7 or equivalent surface dimensional requirements unless otherwise specified in the Ordering Data.

3.9.2 Workmanship.

a. Grinding Defects. Fuel pellets shall be free of grinder induced flats, grooves, or both.

b. Cleanliness. Finished pellet surfaces shall be free of loose fuel powder, pellet chips and foreign materials.

3.9.3 Surface Contaminants. Finished fuel pellet surfaces shall be free of surface contaminants.

3.10 Processes.

3.10.1 Process Qualification. The capability of producing fuel pellets meeting homogeneity figure of merit (3.5.1) plutonium concentration regions (3.5.2) and second phase (3.6) requirements shall be demonstrated. Unless otherwise specified in the Ordering Data, the fuel fabrication process will be considered qualified by producing three consecutive fuel pellet lots meeting requirements for these characteristics. Qualification shall be maintained by verification of compliance with these requirements by periodic testing as specified in the Ordering Data. Failure to meet verification of compliance shall require resampling and requalification. Any change in the fuel pellet fabrication process parameters which may affect fuel pellet homogeneity, plutonium concentration regions, and second phase shall require requalification. Requalification of a qualified fuel fabrication process in which fuel enrichment is the only change shall require production of one fuel pellet lot meeting homogeneity, plutonium concentration region, and second phase requirements.

3.10.2 Fuel Pellet Manufacture. Fuel pellets shall be manufactured by cold pressing and sintering. No material shall be added to the (U-Pu)O₂ except for small quantities of pore formers, organic binders and organic lubricants needed for pelletization.

3.10.3 Lotting and Identification.

a. Fuel pellet lots shall be established for material control, sampling, acceptance, and identification purposes.

b. Fuel pellets within each inspection lot formed for the purpose of acceptance sampling shall be homogeneous in terms of physical, chemical and isotopic properties and shall have been fabricated by the same process. A fuel pellet inspection lot shall be as defined in 1.2.2.

3.10.4 Storage During Processing. Fuel pellets shall be stored in clearly identified containers in a manner to assure conformance with this standard.

3.10.5 Halide Bearing Materials. Fuel pellets shall not be permitted to contact halide bearing materials and shall not be stored in a halide-contaminated atmosphere. (See 6.4.) Materials containing trace amounts of halides shall be qualified before use.

4. QUALITY ASSURANCE REQUIREMENTS

4.1 Quality Assurance Program Requirements. The supplier shall plan, establish, implement, and maintain a documented quality assurance program that fulfills the ANSI/ASME NQA-1 requirements specified in the Ordering Data.

4.2 Quality Assurance Documentation. The supplier is responsible for the preparation of all quality assurance documentation specified in this standard and in the Ordering Data. Records of all tests and examinations shall be kept complete. Quality verification records shall include:

1. The identity of each fuel pellet lot, and all materials as shown on the inspection/test and other quality status records.
2. Reports showing the results of all inspections and tests on pellets and feed materials as required by this standard and applicable material standards.
3. Other data which show the quality status of the fuel pellets and feed materials.
4. Supplier's certification that all fuel pellets meet all requirements of this standard and Ordering Data.

Quality verification records shall be furnished with the delivered fuel pellets.

4.3 Inspection Requirements. Inspection requirements are listed in Table 2. Where "lot qualification" is listed as the inspection plan, the lot shall be accepted only when all of the required number of samples meet the referenced requirement. Where a sampling plan criterion is listed, the supplier shall establish the sampling plan. For those product characteristics requiring acceptance sampling, the supplier shall provide statistically significant evidence, based on his exhibited process capabilities and on the statistical acceptance sampling plan established for use, that the LTPD and β risk specified are not exceeded.

Table 2. Quality Conformance Inspection

Characteristic	Rqmt.	Inspection Plan	Reference Test Method
1. Plutonium isotopic concentration	3.1.1	Verification by analysis of one sample pellet per lot.	NE F 11-1.
2. Fuel pellet fissile concentration	3.2.1	Sampling	Calculate for each fuel pellet lot by Equation 1. (6.3) using feed material isotopic concentration and plutonium concentration for each sample pellet (Ref. Characteristics 1 & 3 and NE E 13-1).
3. Fuel pellet plutonium			
a. Concentration	3.2.2 (a)	Sampling as required for 2 above.	NE F 11-1.
b. Isotopic concentration	3.2.2 (b)	Sampling as required for 2 above.	NE F 11-1.
4. Uranium concentration	3.2.3	Sampling to assure the required confidence interval.	NE F 11-1.
5. Impurity levels			
a. Individual impurities	3.2.4(a)	Sampling	Methods in NE F 11-1.

(continued)

Table 2. Quality Conformance Inspection (Continued)

Characteristic	Rqmt.	Inspection Plan	Reference Test Method
b. Total impurities	3.2.4(b)	Same as a, above	Summation of impurities in (a).
6. Americium Concentration	3.2.5	Same sample taken for 3 (b).	NE F 11-1.
7. Oxygen-to-Metal ratio	3.3	Sampling	NE F 11-1.
8. Gas content	3.4.1	Sampling	NE F 11-1
9. Moisture content	3.4.2	Sampling	NE F 11-1.
10. Homogeneity "Figure of Merit"	3.5.1	<p>Lot qualification:* Three pellets for up to 6 kg of material plus one pellet for each additional 2 kg of material in a sintering batch or in a continuous sintering furnace run.</p> <p>Reduced Sampling Plan:</p> <p>For lots larger than 30 kg, a sample size of 15</p>	<p>Reference (U,Pu)O₂ fuel pellets are to be fabricated by the purchaser to establish Figure of Merit (M) Standards. Such pellets will be thoroughly characterized by electron microprobe in accordance with NE F 11-4 and alpha-autoradiographic analysis in accordance with NE F 11-5. Acceptable and reject reference fuel pellets will be selected on the basis of microprobe analysis, and a Figure of Merit value will be assigned to these. Corresponding alpha-autoradiographs will be produced from these reference pellets and made available as visual stan-</p>

*Or certification of process qualification in accordance with 3.10.1.

(continued)

Table 2. Quality Conformance Inspection (Continued)

Characteristic	Rqmt.	Inspection Plan	Reference Test Method
10. Homogeneity (cont'd)		pellets per lot with C=0 (reference NE F 2-7) may be used.	dards together with the reference pellets. Prepare alpha-autoradiograph of sample fuel pellets in accordance with NE F 11-5. One of the first three sample pellets shall be sectioned and examined longitudinally. The reference pellets shall be alpha-autoradiographed at the same time as each fuel pellet to minimize variation in film properties and development. After comparison with alpha-autoradiograph standards, the manufacturer shall assign a Figure of Merit value to each fuel pellet lot.
11. Plutonium concentration regions	3.5.2	Lot qualification* and reduced sampling plan (same as 10).	Autoradiographic comparison of longitudinal and transverse sections of fuel pellets in accordance with NE F 11-5.
12. Grain size, porosity and secondary phases	3.6	Lot qualification.* Samples selected for 10.	Photomicrographs of pellets prepared in accordance with NE F 11-6.

*Or certification of process qualification in accordance with 3.10.1.

(continued)

Table 2. Quality Conformance Inspection (Continued)

Characteristic	Rqmt.	Inspection Plan	Reference Test Method
13. Fuel pellet (W/L)	3.7	Sampling	<p>Calculate the W/L associated with each sample fuel pellet as follows Sample fuel pellets shall be sintered and shall meet dimensional and surface requirements.</p> <p>1) Measure and record sample pellet length to the nearest 0.0006 in. (0.015 mm).</p> <p>2) Weigh sample pellet to the nearest 0.001 g.</p> <p>3) Calculate the W/L of each sample pellet to the nearest 0.001 g/in. (0.00004 g/mm).</p>
14. Fuel pellet dimensions	3.8		
a. Diameter		All pellets	Laser, micrometer, or limit gaging.
b. Length		Sampling	Micrometer.
c. Dish dimensions-diameter and depth		Process tooling qualification	Visual inspection and comparison with standard derived from punches.
d. Maximum radius or chamfer		Process tooling qualification	Optical comparator.
e. Perpendicularity		Process tooling qualification	Standard gaging.

(continued)

Table 2. Quality Conformance Inspection (Continued)

Characteristic	Rqmt.	Inspection Plan	Reference Test Method
15. Pits, chips, or voids	3.9.1	All pellets	W/L or flaw detection system examination of all pellet cylindrical surfaces and comparison with purchaser-approved physical acceptance standards.
16. Workmanship			
a. Grinding defects	3.9.2 (a)	Sampling inspection at grinder setup and readjustment.	Visual inspection and comparison with purchaser-approved physical acceptance standards.
b. Cleanliness	3.9.2 (b)	Sampling	Visual examination.
17. Surface contaminants	3.9.3	All pellets	Inspection of all cylindrical surfaces by: a. Visual examination and comparison with physical acceptance standards, or b. Surface contaminant detection system.

Procedures for establishment of sample plans are given in NE F 2-7. Reference test methods are acceptable methods. Methods used shall be equal to or better than the methods referenced and shall be approved by the purchaser prior to use.

Where analytical methods are referenced in Table 2 for inspecting for conformance with chemical requirements, other analytical methods may be substituted for the reference test methods. Each analytical method used shall be qualified in accordance with NE F 2-6 and shall be approved by the purchaser prior to use.

4.4 Measuring and Test Equipment.

4.4.1 Environmental Controls. Measuring and test equipment and measurement standards shall be calibrated and utilized in an environment controlled to the extent necessary to assure continued measurements of required accuracy giving due consideration to temperature, humidity, vibration, cleanliness, and other controllable factors affecting precision measurement. When applicable, compensating corrections shall be applied to calibration results obtained in an environment which departs from standard conditions.

4.4.2 Calibration. Measuring and testing equipment shall be calibrated as specified in Basic Requirement 12 and Supplement 12S-1 of ANSI/ASME NQA-1.

4.4.3 Accuracy. Unless otherwise specified in the Ordering Data or approved by the purchaser, determinations shall be made with equipment meeting the requirements of NE F 3-15.

4.5 Tolerance Limit Interpretation.

4.5.1 Chemical Composition. For purposes of determining conformance with chemical composition requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E 29.

4.5.2 Dimensional Requirements. The rounding-off method as defined in ASTM E 29 shall be used for determining conformance with dimensional requirements.

5. PREPARATION FOR DELIVERY

This section does not apply to fuel pellets to be delivered in finished fuel pins.

5.1 Packaging and Shipping. Fuel pellets shall be packaged in a manner to maintain conformance to this standard. A complete description of the packing procedures and shipping containers, including drawings, shall be submitted to the purchaser. Shipping containers shall meet the

requirements in the Code of the Federal Regulations, Title 49, Part 173 (Sections 389 through 398).

5.2 Labeling. Each container shall be clearly marked with radiation warning signs and a label bearing the following information:

1. Purchase order number.
2. Part number (Vendor ID, Drawing No., Revision No., and Item No.).
3. Date packaged.
4. Fissile content.
5. Gross, tare, and net weight.
6. Isotopic content.
7. Fuel pellet lot number.

6. NOTES AND ORDERING DATA CHECKLIST

6.1 Ordering Data Checklist. The following technical and procurement data will be furnished by the purchaser. If not furnished with the purchase documents, the supplier shall request any necessary data before submitting his bid.

<u>Item</u>	<u>Refer to Paragraph</u>
1. Classification (Type)	1.1
2. Other Publications	2.5
3. Drawings	2.6
4. Fuel Pellet Fissile Concentration	3.2.1
5. Plutonium Concentration	3.2.2a
6. Individual Impurities	3.2.4a
7. Total Impurities (limits and order average)	3.2.4b
8. Americium Concentration	3.2.5
9. Fuel Pellet Weight per Unit Length	3.7
10. Dimensions	3.8
11. Pits, Chips or Voids	3.9.1
12. Process Qualification	3.10.1
13. Quality Assurance Requirements	4.1
14. Accuracy	4.4.3

6.2 Fuel Pellet Density Determination. The theoretical density of mixed oxide fuel pellets may be determined from the graph in Fig. 2 which displays the influence of enrichment and oxygen-to-metal ratio on density. Lattice parameters were used as a basis of calculation.

6.3 Fuel Pellet Fissile Concentration Determination. For each fuel pellet lot, calculate fuel pellet fissile concentration using Equation 1.

$$F_1 = 100XY \quad (1)$$

Fig. 2. Theoretical Density of Mixed Uranium-Plutonium Dioxide Sintered Fuel Pellets.

where

F_1 = fuel pellet fissile concentration (percentage ^{239}Pu + ^{241}Pu in fuel)

X = plutonium isotopic ratio (percentage ^{239}Pu + ^{241}Pu in total $\text{Pu}/100$)

Y = plutonium fraction (percentage Pu in total fuel/100)

6.4 Halide Bearing Materials. The use of halide-bearing materials in contact with fuel pellets or for the primary closure of fuel pellet containers is prohibited. Mixed oxide fuel pellets contaminated with chlorine have been known to cause stress corrosion of fuel pin cladding. Localized concentrations of iron and nickel chloride were observed on mixed oxide fuel pellet samples. The chlorine source was polyvinyl chloride used for sealing fuel pellet sample packages. Chlorine produced within sample containers by radiolytic degradation of polyvinyl chloride reacted with iron/nickel particles producing yellow to red colored spots.