TENNESSEE VALLEY AUTHORITY
CHATTANOOGA, TENNESSEE 37401

1040 Chestnut Street Tower II

Mr. John Hickey
Division of Fuel Cycle and
Material Safety
Mail Stop 396-SS
Washington, DC 20555

Dear Mr. Hickey:

RAPID SULFUR METER (RSM) - CONTINUOUS ONLINE NUCLEAR ANALYZER OF COAL (CONAC) BY-PRODUCT MATERIAL LICENSE NO. 41-08165-12 REQUEST FOR AMENDMENT NO. 7

As stated in the Code of Federal Regulations, Title 10, Part 30, Section 30:38, we request that our by-product material license No. 41-08165-12 be amended as indicated below.

Item 7.A - As discussed between Mr. Carrico and Gary MacDonald of my staff, please delete the DOE-SR-CF-307 series sealed source and replace it with DOE-SR-CF-100 series (DOE-SR-CF-001 through 999). The supporting description of the manufacturer, assembly and testing is enclosed for your review. Item 8.A, "not to exceed 30 millicuries per source," will remain the same.

Condition 12 - Due to administrative changes, please delete R. L. Keyser from the list of users and replace with S. J. Stamey.

If you have any questions or require additional information, please call Gary MacDonald at FTS 858-5675.

Sincerely,

John B Brellenthin by JD

John B. Brellenthin, Supervisor
Environmental Support Section

Enclosure

30-19063

FEE EXEMPT

18512

SUSAN JOYCE STAMEY

202 Sequoia Drive
Chattanooga, Tennessee 37411
(615) 624-9878

CAREER OBJECTIVE

Site coordinator for directing the activities of research and development projects.

EXPERIENCE

1978-Present

TENNESSEE VALLEY AUTHORITY, ED&T

Environmental Engineer. Plan, direct, and coordinate field activities for evaluating the operation and efficiency of combustion and pollution control systems. Specific responsibilities have included:

. Directing the operation of a pilot forced-oxidation facility for dewatering limestone slurry from the Widows Creek limestone scrubber. Duties included following startup and coordinating maintenance of the facility; determining specific sampling points and chemical analysis methods; coordinating disposal of the dewatered product; and evaluating operation data and laboratory analysis results.

. Directing gaseous (sulfur dioxide and sulfuric acid mist) and particulate (total and size distributions) emissions tests at the Widows Creek Steam Plant and Scrubber during the following research projects: forced-oxidation, evaluation of turning vane installations on unit 8, and units 1-6 plume studies.

. Directing the testing of a targeted chlorination system for controlling biofoul growth in the condenser at the Kingston Steam Plant. Duties included developing a test plan; following installation of the system and conducting startup tests; providing specifications for prototype sampling equipment; performing routine condenser performance tests; measuring residual chlorine with amperometric titrators and a micro-processor controlled potentiometric analyzer; coordinating maintenance; and evaluating test results.

- Representing TVA as an ASTM collaborator in an EPA/ASTM field evaluation of a proposed method (EPA Method 6B) for determining sulfur dioxide from stationary sources. Duties included selecting and modifying equipment to meet the test method criteria; conducting field experiments on calibration gases and stack gas at the Mongahela Power Plant; analyzed samples for sulfur dioxide, carbon dioxide, and moisture; and calculated sulfur dioxide emission rates.

CURRENT RESPONSIBILITIES

Setting up and directing coal analysis laboratory at the Paradise Coal Wash Plant for analyzing samples from the Process Control research project. Specific duties include specifying equipment and instruments for analysis of coal and refuse from 20 discrete sampling locations in the plant; developing staffing plans and analysis schedules according to project priorities; assisting in the development of a data base; and reviewing and evaluating analysis results.

EDUCATION

B.A. Chemistry, University of Tennessee at Chattanooga
Engineering courses - Thermodynamics, Fluid Dynamics, Heat and Mass Transfer, Fortran and Basic Computer Languages, Numerical Methods

Working toward a Masters in Chemical Engineering

ADDITIONAL TRAINING

Radiological Training Course, TVA, January 1985
Technical Writing, TVA
Fossil Power Plant Operations, TVA
Sampling and Analysis of Utility Pollutants, EPRI
Recent Advances in Pollutant Monitoring, EPA
Source Sampling for Particulate Pollutants, EPA
Workshop on In-Stack Impactors, EPA

DESCRIPTION OF MANUFACTURE, ASSEMBLY AND TESTING

OF SR-CF 100 SERIES ^{252}CF NEUTRON SOURCE

(REVISED 3/84)

A. SCOPE

1. This report describes the manufacture, assembly, and testing of SR-CF-100 series ^{252}Cf neutron sources. These sources are fabricated at the Savannah River Laboratory (SRL) and loaned by the U. S. Department of Energy (DOE) to organizations under contract agreement for approved use in industry. Data and information are provided to show compliance with appropriate regulations.[1-7]

B. REFERENCES

1. Code of Federal Regulations, 10 CFR, Part 20 - Standards for Protection Against Radiation (revised 1979)

2. Code of Federal regulations, 49 CFR, Transportation, Chapter I - Research and Special Programs Administrations, Department of Transportation, Department of Transportation, ¶173.398 (revised 1979).

3. International Atomic Energy Agency, Regulations for the Safe Transport of Radioactive Materials, IAEA Safety Standards, Series 6 (revised 1973).

4. U. S. Atomic Energy Commission, Directorate of Regulatory Standards, Integrity and Test Specifications for Selected Brachytherapy Sources, Regulatory Guide 6.2, Section C.5, 1974.

5. Bureau of Explosives' Tariff, Hazardous Materials Regulations of the Department of Transportation, Specifications for Shipping Containers; ¶178.34, Specification 2R, 1980.

6. American National Standards Institute, Classification of Sealed Radioactive Sources, ANSI N542.1977

7. AEC Manual Chapter 0529-05, Safety Standards for Packaging of Radioactive and Fissile Material.

C. DEFINITIONS

1. SR-CF-100 - a doubly encapsulated neutron source containing up to 10 mg of ^{252}Cf.

2. Filter frit - a small cylindrical filter made from porous platinum used to filter the ^{252}Cf precipitate from the oxalate slurry.

3. Inner capsule - a cylindrical sleeve made from 90% platinum - 10% rhodium used as the primary container for ^{252}Cf material.

4. End plugs - Precision tapered fittings used to seal the ends of the inner and outer capsules.

D. GENERAL DESCRIPTION

The SR-CF-100 is a neutrol source that contains up to 10 mg ^{252}Cf sealed inside a platinum - 10% rhodium inner capsule in turn is sealed in a stainless steel or Zircalloy-2 outer capsule. The source is approximately 1.5 inches long and 0.370 inches in diameter, and one end is threaded so that handling devices can be attached.

1. Identification

SR-CF-100

2. Proposed Use

Applications include:
- Petroleum exploration
- Mineral exploration
- Moisture measurements
- Radiography
- Process control

3. Description of Radioactive Material

The source material is ^{252}Cf oxide (Cf_2O_3) which has been precipated on a platinum filter frit inside the inner capsule. The maximum amount of material in the SR-CF-100 series source is 10 milligrams and the maximum activity is 5.2 curies.

Radiation from ^{252}Cf includes α, β, γ, and neutrons. Alpha radiation is contained within the capsule. β, γ, and neutrons are emitted from the encapsulated source. Beta radiation is very small at distances greater than a few centimeters; the gamma emission rate is 1.3×10^7 photons/sec-microgram and the neutron emission rate is 2.3×10^6 neutrons/sec-microgram. A typical isotopic analysis of the source material is:

Isotope	Atom %
^{249}Cf	0.9
^{250}Cf	11.2
^{251}Cf	3.1
^{252}Cf	84.2
^{253}Cf	0.5
^{254}Cf	0.04
	99.94

4. ANSI Classification[6]

ANSI classification is not applicable for special form radioactive material.

5. Labeling and Instructions for Use of Sources

A ^{252}Cf Source Information Sheet is included with each shipment showing the source strength, closure test method, and test date (Figure 1). In addition, DOE requires that the user have an appropriate state and/or Nuclear Regulatory Commission (NRC) license for handling and use of radioactive material.

Standard industrial SR-CF-100 series sources are identified by an engraved designation SR-CF-001 through 999. Detailed unloading instructions accompany each shipment.

E. ADDITIONAL INFORMATION

1. Pressure Rating

The capsules and various fittings are designed to withstand pressures up to 14,500 psi at 25°C and 6,400 psi at 800°C. This design rating exceeds the full range of internal pressures that could develop during or after the sources are assembled. The two principal sources of internal pressure are discussed below:

a. Expansion of Heated Moisture Trapped Inside Either Capsule

The most likely source of heat to generate this condition is the welding operation wherein the end fittings or plugs are welded to seal the inner or outer capsule. The manufacturing process prior to welding includes temperatures high enough (700°C) to drive off all carbon dioxide and moisture.

No problems have been experienced with weld blowouts which are an indication of trapped moisture expanding from the heat of welding. Also the sealed source is heater to 800°C for 15 minutes prior to conducting the helium leak test.

b. Pressure from Radioactive Decay Products

Radiation decay products of ^{252}Cf will produce helium gas within the capsule. A worst-case calculation for the largest amount of ^{252}Cf (10 mg) at infinite decay indicates the maximum internal pressure would be 183 psia at a capsule temperature of 0°C, and 720 psia at 800°C. Calculated heat transfer and strength characteristics for the SR-CF-100 source are summarized in Tables 1 and 2.

2. **Corrosion Rating**

Because of the excellent corrosion resistance of the 90% Pt - 10% Rh alloy, surface deterioration is insignificant. Long term (1 year) high temperature compatibility tests (800°C) revealed no chemical reaction between the Cf_2O_3 core material and the platinum inner capsule. The welds that seal the inner and outer capsules are of extremely high quality and do not corrode or deteriorate under normal conditions.

3. **Source Life**

The practical service life of the neutron source is determined by the required activity. The source strength diminishes according to the 2.6 years half life of ^{252}Cf. For example, the remaining activity after 5.2 years (2 half lives) would be 25 % of the initial activity.

4. **Current Use of SR-CF-100 Sources**

These sources are now being supplied to universities and industry under contract with DOE for various research projects. They are currently certified by the Department of Transportation (DOT) for not more than 5.2 curies, and renewal of an International Atomic Energy Agency (IAEA) Certificate of Competent Authority has been requested.

F. **MANUFACTURING PROCESS**

1. **General Description of Process**

A filter made of platinum is inserted in the inner capsule and rests on a shoulder as shown in Figure 2. Californium is precipitated from a solution by addition of oxalic acid. The slurry containing californium oxalate is pumped through the inner capsule and the precipitate is deposited on the filter. The sub-assembly is then calcined in a furnace to convert the californium oxalate to Cf_2O_3. Next, tapered end plugs are inserted and sealed by welding. The sealed assembly is shown in Figure 3.

The inner capsule is then inserted in the outer capsule which is a cylindrical member closed at one end, and open at the other, as shown in Figure 4. A tapered plug is inserted in the open end and sealed by welding. This completes the outer capsule assembly.

h. Verify that the surface is free of contamination by smear tests. The alpha count must be less than 50 d/m, which corresponds to 0.02 nanocuries or less of removable contamination.

i. Place the inner capsule in the outer capsule and insert the end plug.

j. Seal with a tungsten-arc weld.

k. Assay for ^{252}Cf content.

l. Perform a helium leak test on the outer capsule.

(1) Externally pressurize with helium at 300 psi.

(2) Measure helium in the system (the equipment is calibrated to detect a helium loss of less than 2.8×10^{-8} cm^3 helium per second).

m. Verify that the surface is free of contamination by smear and leach tests. The alpha count must be less than 10 d/m, which corresponds to 0.0135 nanocuries or less of removable contamination.

G. QUALITY CONTROL OF THE SOURCE FABRICATION PROCESS

Each source is given all of the following checks during construction:

1. Components and tubing used to make the inner and outer capsules are inspected with a stereoscopic microscope. Materials containing seams or folds are rejected.

2. Components and tubing are gauged for conformance with mechanical specifications.

3. Inner and outer capsule end welds are inspected with a 20X stereoscopic microscope. Misshapen or irregular welds are rejected.

4. The inner capsule closure weld is inspected with a periscope. Misshapen or irregular welds are rejected.

5. The diameter and length of the inner and outer capsules are measured.

6. A helium leak test is performed on the inner capsule. Sources with helium leaks greater than 2.8×10^{-8} cm^3/second are rejected.

7. The outer capsule closure weld is inspected with a periscope.

8. The ^{252}Cf content is measured with a precision neutron counter.

9. A helium leak test is performed on the outer capsule. Sources with He leaks greater than 2.8×10^{-8} cm/^{3} second are rejected.

10. Acceptable surface decontamination is verified by smear test. The limit is 0.0135 nanocuries of removable surface contamination which is weld below the limit of 0.5 nanocuries of removable contamination specified by Regulatory Guide 6.2.

11. Quality control for the californium is performed by measurement of the neutron emission rate of an aliquot of the starting material and by analyzing for isotopic content and chemical purity. The neutron emission rate is assayed by measuring the ion current from ^{235}U fission counters surrounding the source in a polyethylene moderator. Isotopic content is measured by mass spectrometry, and chemical purity is determined by spark source mass spectrometry.

H. TESTING AND EVACUATION METHODS

1. Inner Capsule

The basic test to determine the integrity of the inner capsule is the Helium Leak Test. The sealed capsule is pressurized in 300 psi helium for 30 minutes. Leak tests are performed on individual capsules in a helium leak detector whose lower detection limit is 2.8×10^{-8} standard cubic centimeters of helium per second. All capsules must show no detectable leak. Following the leak tests, the capsule is decontaminated to a level of less than 50 d/m $\alpha - \gamma$ transferable radioactivity as determined by a wipe test.

2. Outer Capsule

The integrity of the outer capsule is tested with a helium detector.

I. PROTOTYPE EVALUATION

The first sources fabricated in the SR-CF-100 series were subjected to a variety of tests to prove their integrity. The purpose was to demonstrate the adequacy of both the source design and method of manufacture. All of these tests equaled or exceeded the IAEA requirements.[3] After each test, the source was visually inspected for damage, and the integrity of the seal was determined using a helium leak test capable of detecting a loss of helium of less than 2.8×10^{-8} cm^3 per second.

1. **Free Drop**

The source was dropped through a distance of 30 feet onto a flat, essentially unyielding horizontal surface, so as to suffer maximum damage. No structural damage or loss of containment resulted.

2. **Percussion**

The capsule was placed on a one-inch thick sheet of lead, hardness number 3.5 to 4.5 on the Vickers scale, supported by a smooth, essentially unyielding surface. A one-inch diameter steel rod weighing three pounds was dropped from a height of 40 inches so that the flat end impacted the test capsule. No loss of containment resulted.

3. **Heating**

The source was heated in air to a temperature of 800°C and was held at that temperature for a period of 10 minutes. No damage or loss of containment resulted.

4. **Immersion**

The source was immersed in water at room temperature for 24 hours. The water pH was 6-8, with a maximum conductivity of 10 micromhos/cm. The water and source were heated to a temperature of 50° + 5°C and maintained at this temperature for 4 hrs. No damages or loss of containment resulted. The source was stored for 7 days in still air at a temperature of 30°C. The source was immersed in water at room temperature for 24 hours. The water pH was 6-8, with a maximum conductivity of 10 micromhos/cm. No damage or loss of containment resulted.

J. **MECHANICAL TESTS**

The integrity of the source construction and seal weld was demonstrated by sucessfully subjecting the secondary capsule to internal and external pressures far in excess of pressures expected under the most adverse service conditions. Infinite decay pressure created in the inner capsule from alpha decay and fission gas buildup in a 10 mg source is calculated to be 183 psi at standard temperature, or 720 psi at 800°C. Calculated heat transfer and strength characteristics for the SR-CF-100 source are summarized in Tables 1 and 2.

1 **Burst Tests of Circumferential Weld**

Hydrostatic burst tests on the circumferential closure weld in the outer capsule revealed that the average burst strength of the circumferential weld is 52,000 psi for stainless steel and 41,000 psi for Zircaloy-2 at 25 C. At 800 C the calculated burst strength for stainless steel is 2800 psi and 10,000 psi for Zircaloy-2 (internal pressure).

2. **External Loading Tests**

The two worst conditions to which the source is likely to be subjected are: a) crushing by a heavy object such as the shipping cask; and b) collapse under hydrostatic pressure, such as the capsule would experience in a deep-wall or deep-sea environment.

a. Crush Test

A large shipping cask for several milligrams of ^{252}Cf may weigh as much as 20 tons. Assuming half the weight of the cask might come to rest on the capsule, prototype sources were placed between stainless steel anvils loaded with a total of 10 tons, removed and pressurized with helium at 300 psi for 30 minutes, then tested for leaks with a helium leak detector. At a lower detection limit of 2.8×10^{-8} standard cubic centimeters of helium per second, no leaks were detected.

b. Hydrostatic Compression Test

The hydrostatic pressure at 10 miles dept in a bore hole is about 25,000 psi. Test capsules were subjected to 25,000 psi helium pressure without measurable deformation, and then tested for leaks with a helium leak detector whole lower detection limit is 2.8×10^{-8} standard cubic centimeters of helium per second. No leaks were detected.

TABLE 1

Calculated Heat Transfer and Strength Characteristics of 100 Series Capsules

Inner Capsule Material	90% Pt - 10% Rh
Source strength, mg ^{252}Cf	10
Heat generation, watts	0.4
Adiabatic temperature rise of inner capsule, °C/min	19
ΔT between capsule and air, °C	62
Gas pressure in inner capsule after infinite alpha decay	
at 0°C gas temperature, psi	183
at 25°C gas temperature, psi	200
at 800°C gas temperature, psi	720
Rupture pressure in inner capsule	
at 25°C capsule temperature, psi	14,500
ata 800°C capsule temperature, psi	6,400

Outer Capsule Material	Zircaloy-2	304L SST
Gas pressure in outer capsule if inner capsule leaks after infinite decay		
at 0°C gas temperature, psi	78	78
at 25°C gas temprature, psi	86	86
at 800°C gas temperature, psi	308	308
Rupture pressure in outer capsule		
at 25°C capsule temperature, psi	17,900	23,800
at 800°C capsule temperature, psi	5,900	2,300

TABLE 2

Properties of Capsule Materials

Alloy	Tensile Strength, psi at Temp. 25°C	800°C	Density, g/cc	Heat Capacity, cal/g-°C
90% Pt - 10% Rh	45,000	20,000	19.97	0.034
90% Pt - 10% Ir	55,000	35,000	21.53	0.031
Zircaloy-2	60,000	20,000	6.55	0.077
304L SST	80,000	8,000	8.02	0.12

FIGURE 1. Information Sheet

"Special Form" - ^{252}Cf Neutron Source

Source Identification Number ______________________ Date: ________________

Shipped To: ____

____

Specification: Secondary Capsule Drawing ______________________

Primary Capsule Drawing ______________________

DECONTAMINATION AND CLOSURE TESTS

Method

Capsule surfaces are decontaminated after closure in an ultrasonic bath until the flush solution contains less than 200 d/m per milliliter total alpha activity. The capsule is further decontaminated, if necessary, until all exterior surfaces are free of contamination (<9 d/m alpha and 10 c/m beta-gamma) as determined by a wipe test. Each assembly is immersed in a helium atmosphere with a pressure of at least 300 pounds per square inch for a period of 30 minutes, then transferred to a helium leak detector. The leak detector has a minimum sensitivity of 2.8×10^{-8} standard cubic centimeters of helium per second.

Tests

The finished capsule was found free of detectable leaks and contamination on ______________________.

CALIFORNIUM CONTENT

Assay

The neutron emission rate of the finished assembly is determined by comparing its strength to that of a ^{252}Cf source calibrated at the National Bureau of Standards. The comparison is made by inserting the capsule assembly in an array of fission tube counters and measuring the subsequent induced electric current by a sensitive ammeter. The ^{252}Cf content given below is the effective or net californium content calculated from the neutron emission rate and is given in equivalent weight units assuming 2.311×10^6 neutrons per second per microgram of ^{252}Cf. Corrections are made when necessary for the ^{254}Cf contribution to the total neutron emission rate. The ^{252}Cf present is assumed to decay with an effective half-life of 2.646 years; the ^{254}Cf, if present, is assumed to decay with a 60.5 day half-life.

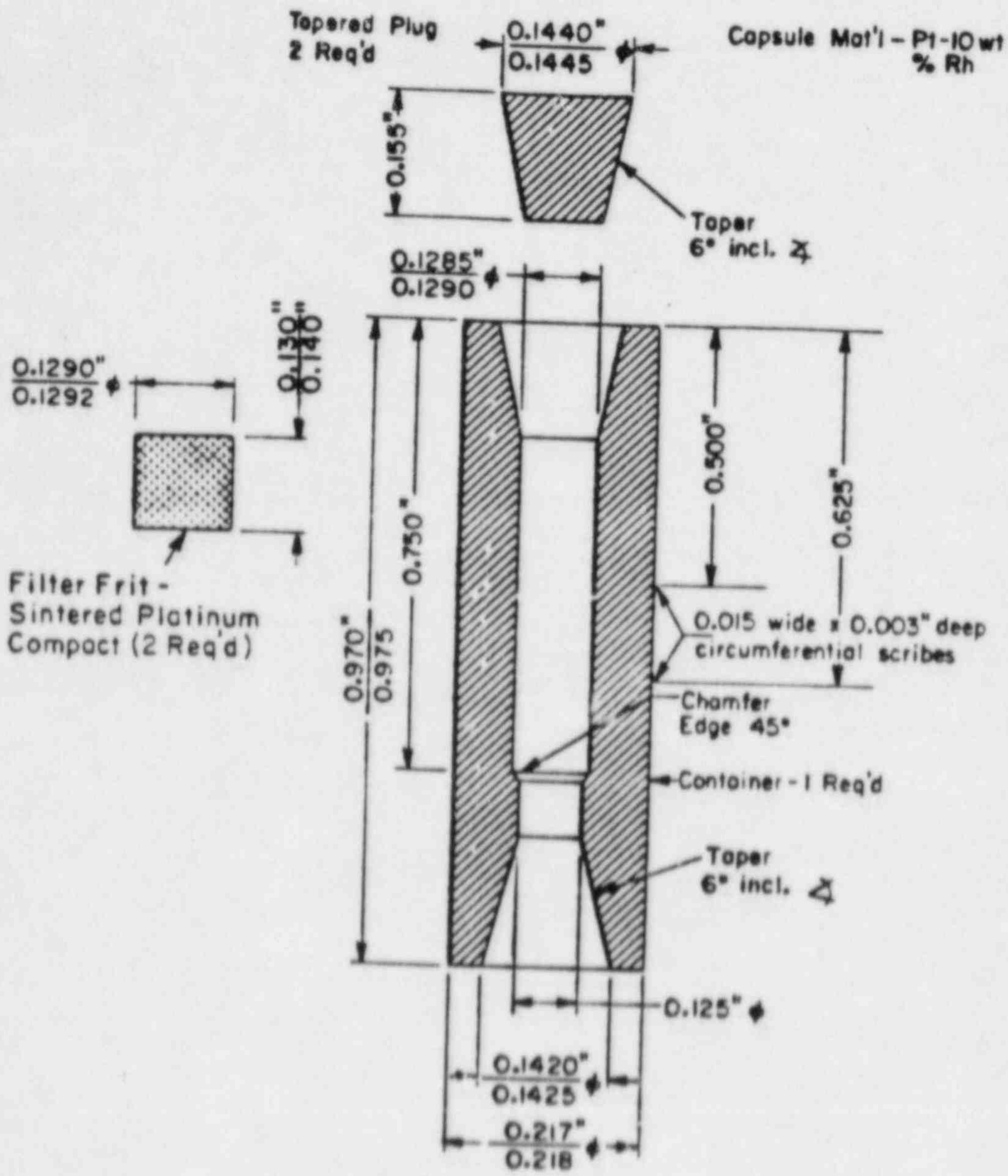
Contents

The total neutron emission rate of this source was found to be ____________ neutrons per second with a standard error of $\pm 3.0\%$ on ______________________. The ^{254}Cf contribution to the total was calculated to be ______________ per second on the same date. The effective ^{252}Cf content was calculated to be ______ µg equivalent with a standard error of $\pm 3.0\%$.

The radiation intensity at three meters from a source in air at standard atmospheric conditions and without contributions from scattering media is no greater than 400 mrem/hr neutrons plus 30 mR/hr γ for each milligram of ^{252}Cf in the capsule.

The fabrication and encapsulation costs excluding the value of the ^{252}Cf are ______________.

____
W. S. Curlee
Laboratory Services Division
Savannah River Laboratory
E. I. du Pont de Nemours and Co.



SK-183-PM

FIGURE 2. SR-CF 100 Series - Inner Capsule Components

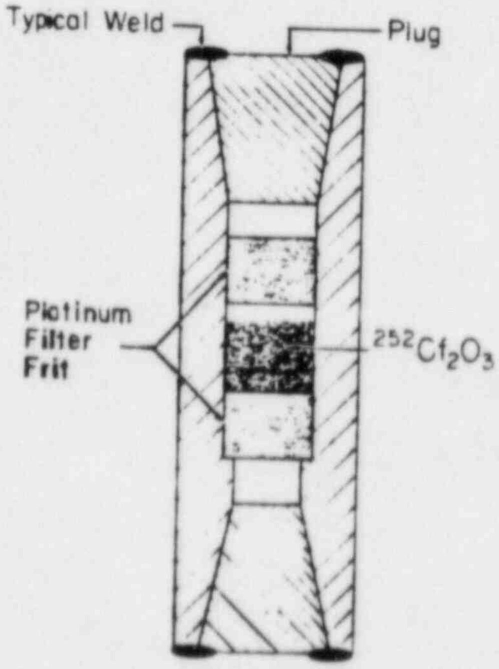


FIGURE 3. Completed Inner Capsule Assembly

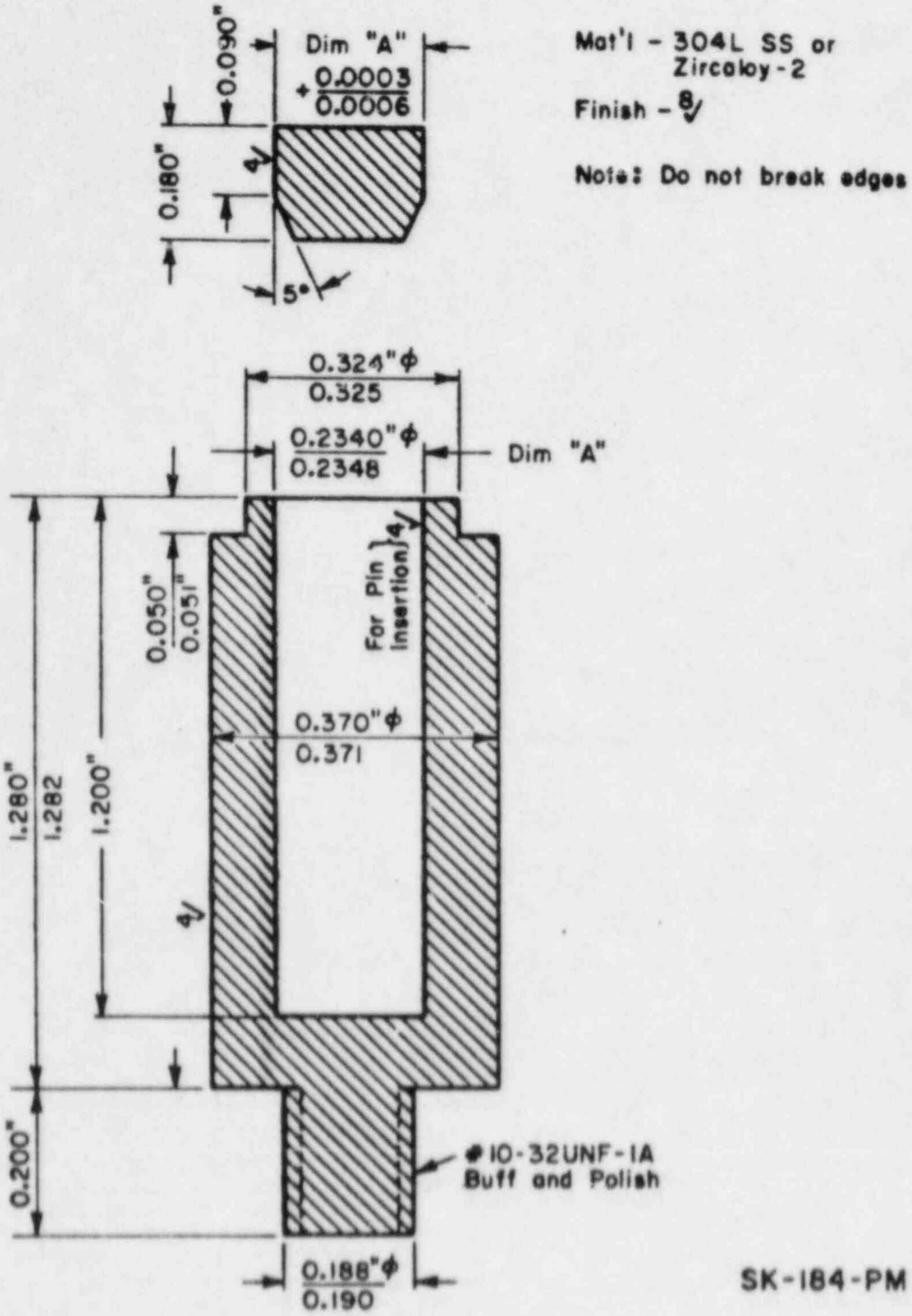


FIGURE 4. SR-C1-100 SERIES - SECONDARY CAPSULE

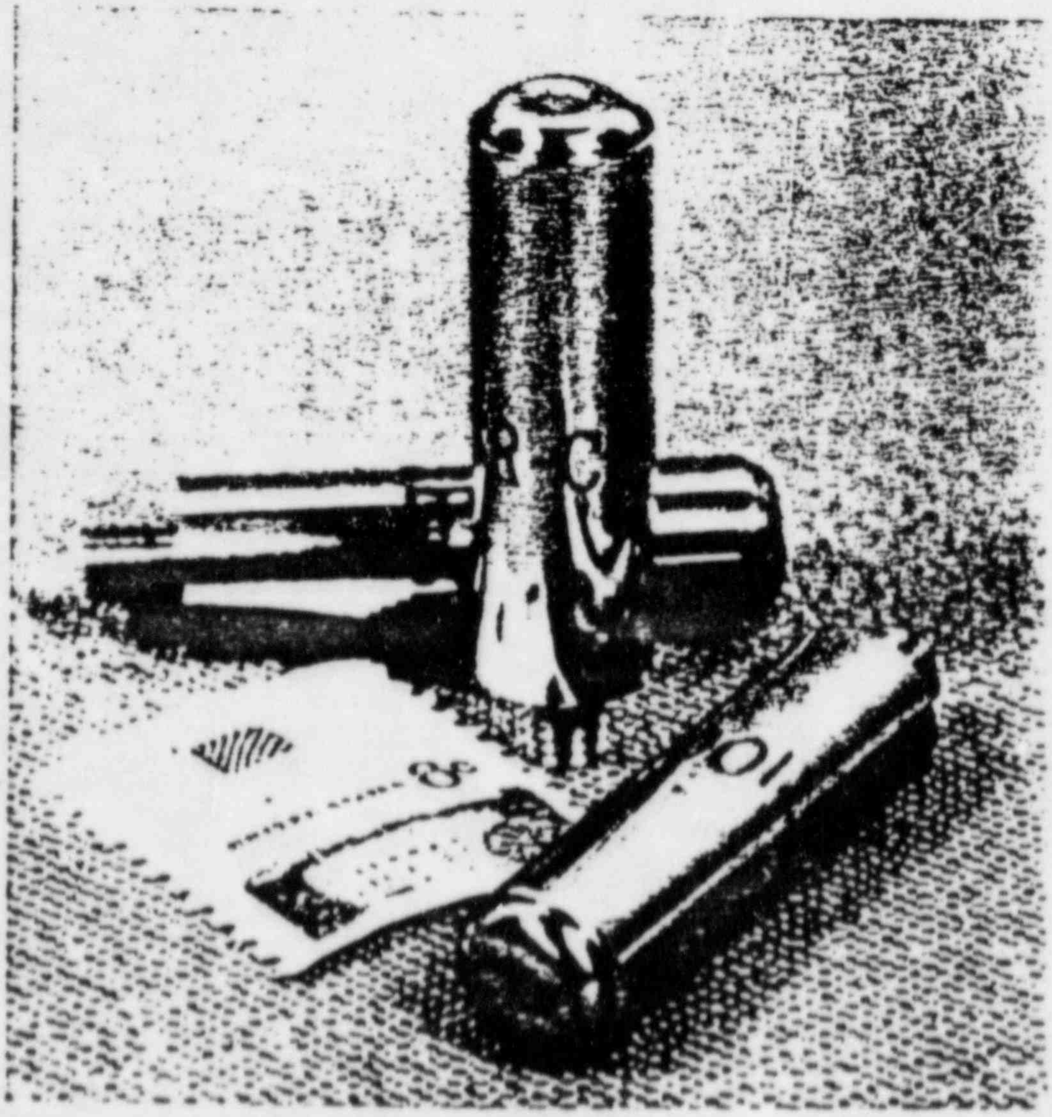


FIGURE 5. Outer Container Identification