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MARK 53 FABRICATION PROCESS DESCRIPTION FOR INITIAL PRODUCTION

INTRODUCTION

This letter documents billet and tooling designs and recommends fabrication conditions for initial production of the Mark 53 Np target tube developed for replacement of the present Mark 53 design.(1) This information has been discussed with J. P. Maloney (300-M) and R. F. Rogers (200-F).

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The process description is based on experience gained from fabrication and evaluation of twenty-nine test elements using ThO_2 and depleted UO_2 as a stand-in for NpO_2. Initial production of NpO_2 containing tubes will serve as a final process demonstration. Fuel and Target Technology will continue to evaluate the process and SRL will continue to provide technical support. SRL will issue a technical manual and incorporate necessary changes based on continued evaluation.

SUMMARY

A process is recommended for NpO_2-Al core billet preparation and extrusion into Mark 53 tubes. Production experience may indicate desirable changes in the recommended process, but these modifications should not be incorporated into production until the possible effects on tube quality are evaluated.

Billet design, identification of available tooling, and fabrication conditions and techniques are presented in Tables I through VII and in Figures 1 through 5.

DISCUSSION

General

The Mark 53 target design offers the following improvements over the current Mark 52:[1]

- The assembly is sized for irradiation in universal sleeve housings.
- The cold compacted cores are shaped for improved core uniformity.
- NpO_2 concentration has been reduced to accommodate the hardening effects of the NpO_2-Al reaction.

The Mark 53 is nominally 3.700-inch OD with 0.213-inch wall thickness and has a 0.125-inch-thick cylindrical core. Uniform core thickness is obtained from use of 16 shaped, segmented compacts in a ring to form a continuous core in the extrusion billet. A double ring of compacts (butted end to end) is used to form billet cores for tube cores over 5 feet long. Nominal core loading will be 150 grams of Np/foot.

The basic steps of the fabrication process are shown in Figure 1. They are identical to those used for production of current Mark 52[2] and Mark 61[3] Np targets and include:

1. blending and cold compaction of NpO_2 and Al powders into core compacts.
2. core loading, welding, leak checking, and outgassing of billets.
3. hot extrusion and cold drawing of tubes.

Changes in the Mark 52 fabrication conditions recommended for the Mark 53 are based on development tests aimed at improving the homogeneity of the compacted core and reducing the extent and effect of the NpO_2-Al reaction. These changes are incorporated into the discussion of each phase of the process.

The process description is based on experience gained from fabrication and evaluation of twenty-nine test elements using ThO_2 or depleted UO_2 as a stand-in for NpO_2. The stand-in cores were outgassed at normal outgassing temperature for extended times to approximate conditions required for hard particle formation which occurs at shorter times in the more reactive NpO_2-Al cores. Because NpO_2 is more reactive, the extent and effect of hard particle formation may be significantly greater than experienced with ThO_2 and UO_2 stand-in. Therefore, the initial production of NpO_2 containing tubes will serve as the final process demonstration.

Core Fabrication

Feed Materials - NpO_2 (standard HB-line product) powder should be screened through 100 mesh U. S. Standard Sieve prior to blending. This will remove particles or agglomerates (including possible foreign inclusions) larger than .006 inch. This specification is currently applied to the Al powder (Essential Material Specification No. 87).

Powder Blending - Tests of the homogeneity of blends for Mark 52 production indicate that present blend technique is adequate. After blending, the Al and NpO_2 powders will tend to segregate (due to size and density differences) if they are mechanically agitated. Manual handling of blends should be gentle and minimized. Powders should be compacted the same day that they are to be blended.

Tooling Lubrication and Die Filling - The Al-stearate-dodecanol mixture (Table I) used in Mark 52(4) and Mark 61(3) production is recommended for lubricating compaction punches and dies. The combination die loader-lubricant applicator tool, Figure 3, provided for use in the Mark 53 compaction die is a modification of the similar tool developed for Mark 61.(3) Leather was substituted for the cloth-covered sponge used on the Mark 61 tool because the strength of the sponge-fill tube bond was found to be inadequate during Mark 61 production. The Mark 53 lubricator should be evaluated with Al powder compacts and Building 235-F production conditions before use in production of NpO_2-Al compacts. The use of this tool in the Mark 53 compaction die is described in Appendix I.

Compaction - Core compact density was lowered from 87 (Mark 52) to 80 percent of theoretical density to reduce the deformation of the aluminum powder and destruction of the Al_2O_3 surface film on the aluminum particles. This film serves as an effective diffusion barrier to the NpO_2-Al reaction. Regardless of the compaction density, the density after extrusion becomes approximately 100 percent. However, at lower compaction densities, the tube core becomes thinner during extrusion than at higher compaction densities.

Extruded core lengths are predictable from compact core lengths and compact densities using the empirical relation:

$$\text{Tube Core Length} = \frac{\text{Compact Core Length}^{*}}{5.782} \times (0.592 \times \text{Compact Density in \%} + 48.64).$$

Data from nine development tubes correlated 95% with this formula.

Billets for extrusion into 4 or 5-ft-long cores contain a single ring of compacts; for core lengths greater than 5 ft, two rings of compacts, butted end to end, are required. Front and rear compacts have the same shape (Figure 2). The compact lengths and volumes required for 4, 5, 8, and 10-ft core tubes are given in Table II. As recommended for Mark 61 core compaction,[3] compact length should be controlled by length measurement while the compact is still in the die. Desired changes in core length should be made by changing the quantity of aluminum in the charge while maintaining compact density. Changing the billet core length by changing the compact density at constant compact weight has little effect on extruded core length.

Expected compaction forces based on compact density of 80 percent are given in Table II. With the lubrication techniques used, ejection force for UO_2- and ThO_2-Al compacts was approximately 20 percent of compaction force.

Billet Design and Assembly

The Mark 53 billet was designed to reduce the probability of weld failure and subsequent tube blistering during extrusion. The welded front and rear rings are supported by shoulders on the inner and outer sheaths, Figure 2. This design restricts relative motion between the rings and sheaths and reduces the shear stress acting on the welds during billet upset. A nominal 0.100-inch gap is provided in the total length of the front-plug-core-rear-plug stack to allow within-tolerance variations in component length without disturbing the desired weld ring-sheath shoulder contact.

Procedures for billet assembly and welding are similar to Mark 52 procedures. The lower-density Mark 53 compacts are more fragile than Mark 52 compacts and require careful handling during inspection and billet assembly. In loading billets with tandem rings of compacts, the front and rear compacts should be matched to minimize deviations from nominal core length.

A method for leak checking welded billets at elevated temperatures was developed and is recommended for the Mark 53 process. All Mark 53 and Mark 61 test billets (a total of 45) were leak tested by this new method

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* Adjusted for special end shapes. For the compact shape recommended (Figure 2), this is the length measured on the OD or ID.

after passing a bubble test inspection. Approximately 20 percent leaked and were accepted for extrusion only after being repaired and passing the hot leak check; no tubes were blistered in test extrusions of either the Mark 61 or Mark 53 development programs. The technique and facilities required for this leak check are described in Appendix II.

It is recommended that C-20 (extruded 356 Al alloy, EMS 23) end plugs be used for the 18.55 wt % Np (21.05 wt % NpO_2) cores (corresponds to 150 g Np/ft of extruded tube core).

Coextrusion

Test extrusions were made in Buildings 320 and 321-M with ThO_2-Al or depleted UO_2-Al billet cores. Only two of the billets were preheated with the half-cylinder steel pan recently specified for Mark 53 Np billets.[5] To evaluate the effect on tube quality of hard particles formed by the reactive NpO_2-Al system, the less reactive ThO_2-Al test cores were held at billet outgassing temperature (525°C) for up to 150 hours rather than the recommended 16-32 hours. Hard particles developed in the ThO_2 cores but cladding thickness over the core and end defects was not significantly affected. The FDA has not been calibrated for Mark 53 tubes but measurements using substitute aluminum standards indicated approximate density ratios up to 1.18 times nominal in 1/4- x 1-inch areas.

Billet Outgassing - Previous comparison of ThO_2-Al and NpO_2-Al cores in Mark 52 billets revealed that Np cores require longer outgassing times to reach desired leak rate. Therefore, the outgassing conditions suggested for initial Np billets, Table III, were derived from experience in current Mark 52 and Mark 61 production.

Billet Preheat - Billet preheat conditions for Building 321-M are given in Table IV. These conditions were determined in tests with a ThO_2-Al core billet fitted with thermocouples and heated in half-cylinder steel pans. Preheat conditions for Building 320-M were not determined because the largest available heating coil could not accommodate the billet inside the prescribed metal pan.[5] When a suitable coil is available and before extruding Mark 53 tubes in 320-M, preheat tests will be made with a billet containing thermocouples.

Extrusion - Recommended extrusion conditions are given in Table V. The container, die, and mandrel should be thoroughly cleaned between each extrusion to minimize rejection due to surface defects.

Drawing and Finishing - Standard station procedures for Building 321-M are satisfactory for tube cleaning, drawing,[6] and stretch straightening.[7]

SRL will assist in the inspection of the first Np elements; reject tubes will be destructively evaluated. Close adherence to the recommended fabrication process and accurate documentation of actual conditions will permit a more quantitative evaluation of the process and will provide a basis for process modifications if needed to improve tube quality.

The information in Figures 1 through 5, Tables I through VII, and in the Appendices provides tooling and billet designs and fabrication techniques resulting from the Mark 53 development program. Changes deemed necessary must be tested to evaluate effects on the final product before they are adopted as part of the production process and incorporated in the Technical Manual.

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By: C. L. Selby

CLS:ehj

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1. R. A. Gregg to W. P. Bebbington, ^{237}Np Target Tube Development, DPST-69-443, July 11, 1969 (U).

2. ^{237}Np Compact Billet Fabrication - Technical Standards, DPSTS-235-F-NC, March 12, 1969 (S).

3. R. A. Gregg to W. P. Bebbington, Mark 61 Target Fabrication Process Description for Initial Production, DPST-70-277, March 11, 1970 (S).

4. J. B. Mellen and J. R. Nauright, TA 2-689/3-932, DPSOX-7392, Alternate Die Lubricants for Np Compacts, August 14, 1969 (U).

5. J. R. Nauright and J. B. Mellen, Development and Production of Mark 53 Neptunium Oxide Target Tubes, TA 3-950/2-712, DPSOX-7531, March 23, 1970 (C).

6. 321-M Station Procedure for Drawing and After-Drawing Cutoff (Rev. 11), April 14, 1967.

7. 321-M Station Procedure for Stretch Straightening (Rev. 10), February 9, 1966.

TABLE I

TOOLING AND BILLET LUBRICATION

I. Compaction die and tooling

Lubricant - 15 wt percent aluminum stearate[1] in dodecanol[2]

Temperature - maintain dodecanol as liquid (M. P. ~70°F)

II. Billet

After outgassing and prior to preheat, dip billet in "Aquadag"[3]

Billet temperature - 100°C

Lubricant - 20 parts water to 1 part "Aquadag" (by volume)

III. Extrusion

After preheat and prior to extrusion, a lead-oil mixture is to be liberally applied to:

Applied to	Lubricant
Billet Cut-off Block Dummy Block Container Die Mandrel	Lead-oil - 1 part lead to 1 part oil by volume[4]

1. WITCO No. 18, Whitco Chemical Corp.
2. Technical Grade, EMS 90
3. "Aquadag" - Trademark of Acheson Colloids Co. - A collodial graphite suspension
4. Du Pont Code A-408

TABLE II

COMPACT DIMENSIONS AND COMPACTION FORCES

Tube Core Length, ft	Compact Length, inches	Compact Volume, cc	Compaction Force, lb
4 and 8*	3.400 ± .050	78.7	27,750
5 and 10*	4.126 ± .050	98.4	29,000

* Two rings of compacts butted end to end

TABLE III

BILLET OUTGASSING

Temperature (°C)	525
Time (hours)	24 (min), 28* (max)
Rate of Pressure Rise (leak rate, 3 min)	$<$ 300 μ Hg

* If desired leak rate is attained at any time between 24 and 28 hours, it is recommended that the furnace temperature be set at $<$ 200°C and evacuation continued until billets are ready for extrusion.

[redacted]

TABLE IV

BILLET PREHEAT
(Building 321-M)

Coil Length (inches)	14.5
Coil Diameter (inches)	9
Tap Setting	3
Distance Billet inside Coil (inches)	4-1/2
Power Setting	2-B
Voltage	46
Controller Setting	425-450
Time (minutes)	
Preheat, minimum	6
Soak, minimum	9
Preheat to Extrusion maximum	1
Billet Temperature* (°C)	380-420

* As determined by contact pyrometer on OD.

[redacted]

TABLE V

EXTRUSION CONDITIONS

	Temperature[1] (°C)
Billet	400 ± 20
Die	300 ± 30
Container	350 ± 30
Mandrel	300 ± 60
Cut-off Block	300 ± 30
Dummy Block	300 ± 60

	Ram Force[2] (tons)	
	Nominal	Maximum
Core	800	900
Rear Defect	850	1050

1. Temperature of working surface as determined by contact pyrometer.
2. Typical values observed for $Al-ThO_2$ and $Al-UO_2$ (depleted) billets.

TABLE VI

FABRICATION TOOLING

		Part Number	Drawing Number
I.	Compaction		
	Die	ST-PM-996	ST-PM-996
	Punches	ST-PM-996	ST-PM-996
	Loader-Lubricator	ST-PM4-984	ST-PM4-984
II.	Extrusion		
	Container	SO-NS 8-1/4"	-
	Stem Size	8.000" OD, 4.564" ID	-
	Dummy Block	5708-1 5708-2	ST-PM3-1047
	Die	5706-1 5706-2	ST-PM3-1049
	Mandrel	SRL-6908-1	ST-PM4-853
III.	Drawing		
	Die	5709-1 5709-2 5484	ST-PM3-1048 ST-PM3-1037
	Mandrel	SRL-D-6908-1	ST-PM3-854 (3)*
IV.	Stretch straightening		
	Mandrel	ST-PM3-933	ST-PM3-933

* Latest revision

TABLE VII

MARK 53 TARGET DRAWING INDEX

Billet Component Drawings

	Drawing No.	
Assembly	ST-PM4-891	(23)*
Outer Sheath	-990	(13)*
Inner Sheath	-990	(13)*
Front Ring	-991	(1)*
Evacuation Ring	-991	(1)*
Front Plug	-959	(8)*
Rear Plug	-959	(8)*
Core	-996	(5)*
Cut-off Block	ST-PM3-1070	

* Latest revision

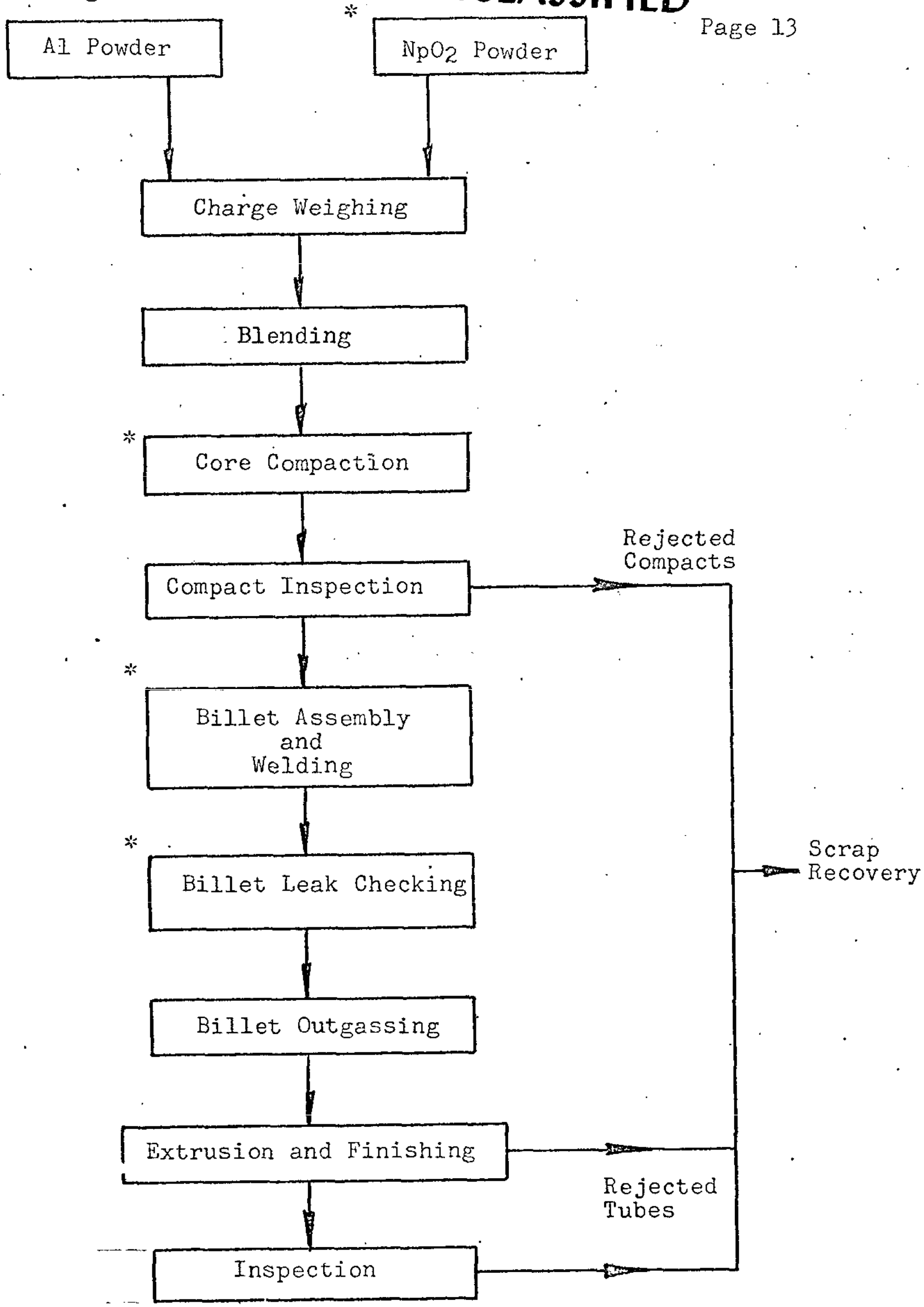

FIGURE 1. MARK 53 FABRICATION PROCESS

* Conditions or technique significantly different from Mark 52 process.

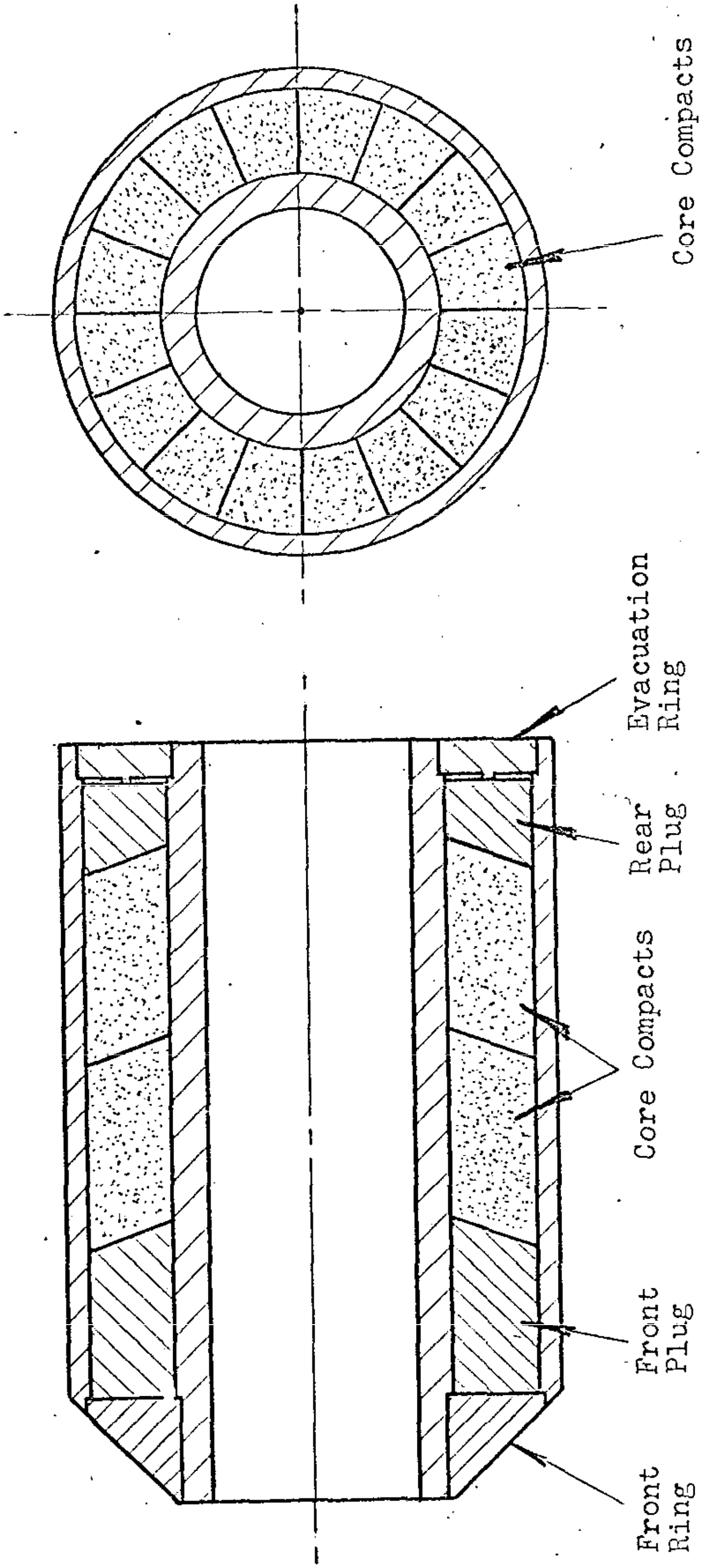

FIGURE 2. LONGITUDINAL AND TRANSVERSE CROSS SECTIONS OF MARK 53 BILLET WITH TANDEM RINGS OF COMPACTS FORMING CORE. Ref. ST-PM4-891

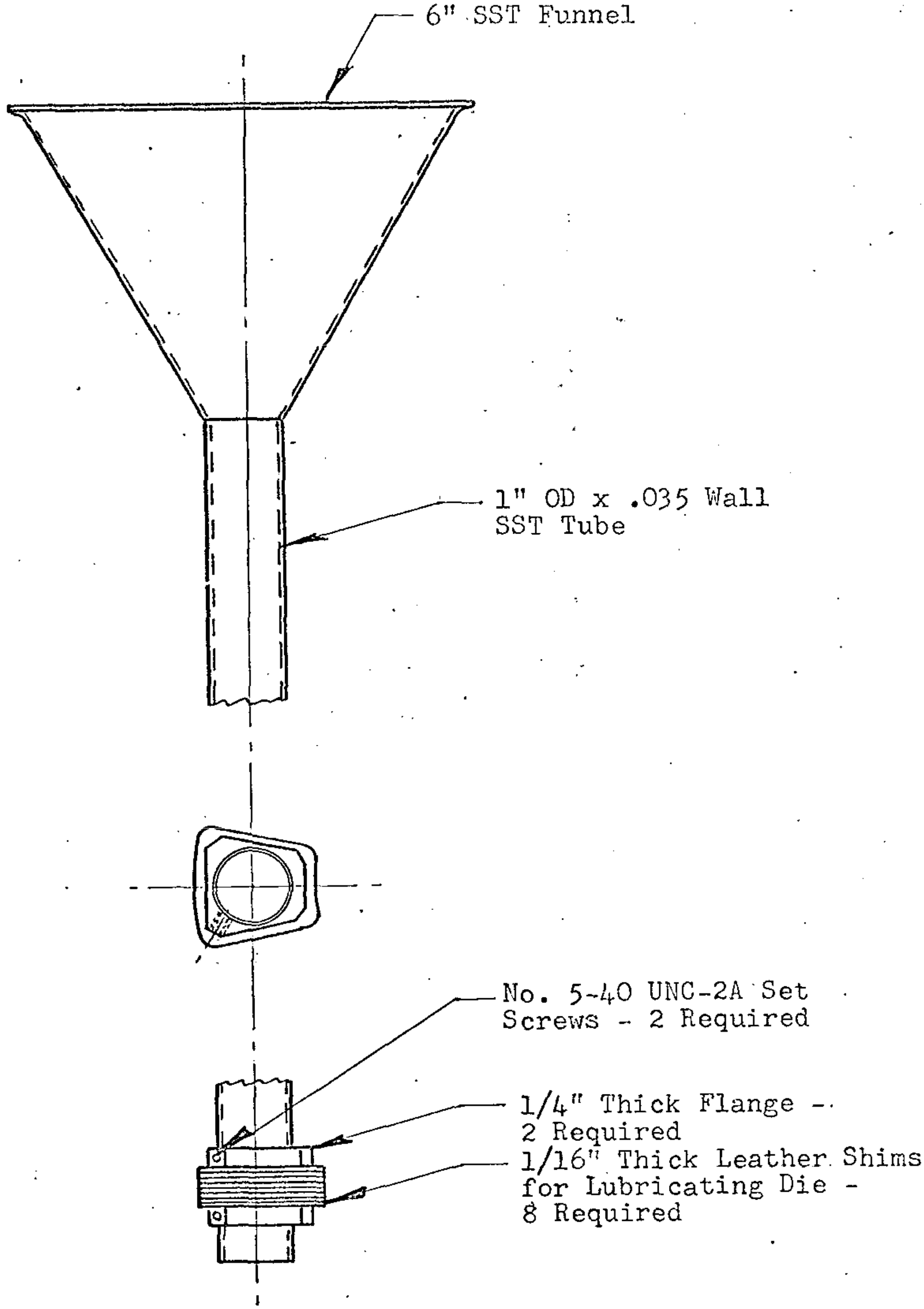

FIGURE 3. DIE LOADER-LUBRICATOR TOOL FOR MARK 53 COMPACTION DIE. Ref. ST-PM3-1059

APPENDIX I

USE OF COMBINATION DIE LOADER-LUBRICATOR TOOL

A sketch of the die loader-lubricator tool is shown in Figure 3 and its use is demonstrated in Figure 4 (a) and (b). Before inserting the tool into the die cavity, apply lubricant only to the leather applicator. Keep the remainder of the tool as dry as possible, particularly the inside of the fill column. Lower the tool into the die, insert the bottom punch and fill the column with the powder charge through the funnel, Figure 5 (a). Slowly withdraw the tool, Figure 5 (b); this will apply lubricant to the die walls while filling the die with powder.

To prevent inclusion of debris in the powder compact, the physical integrity of the tool should be visually assessed after each compaction. Any sign of deterioration should be immediate cause for substitution of a new tool.

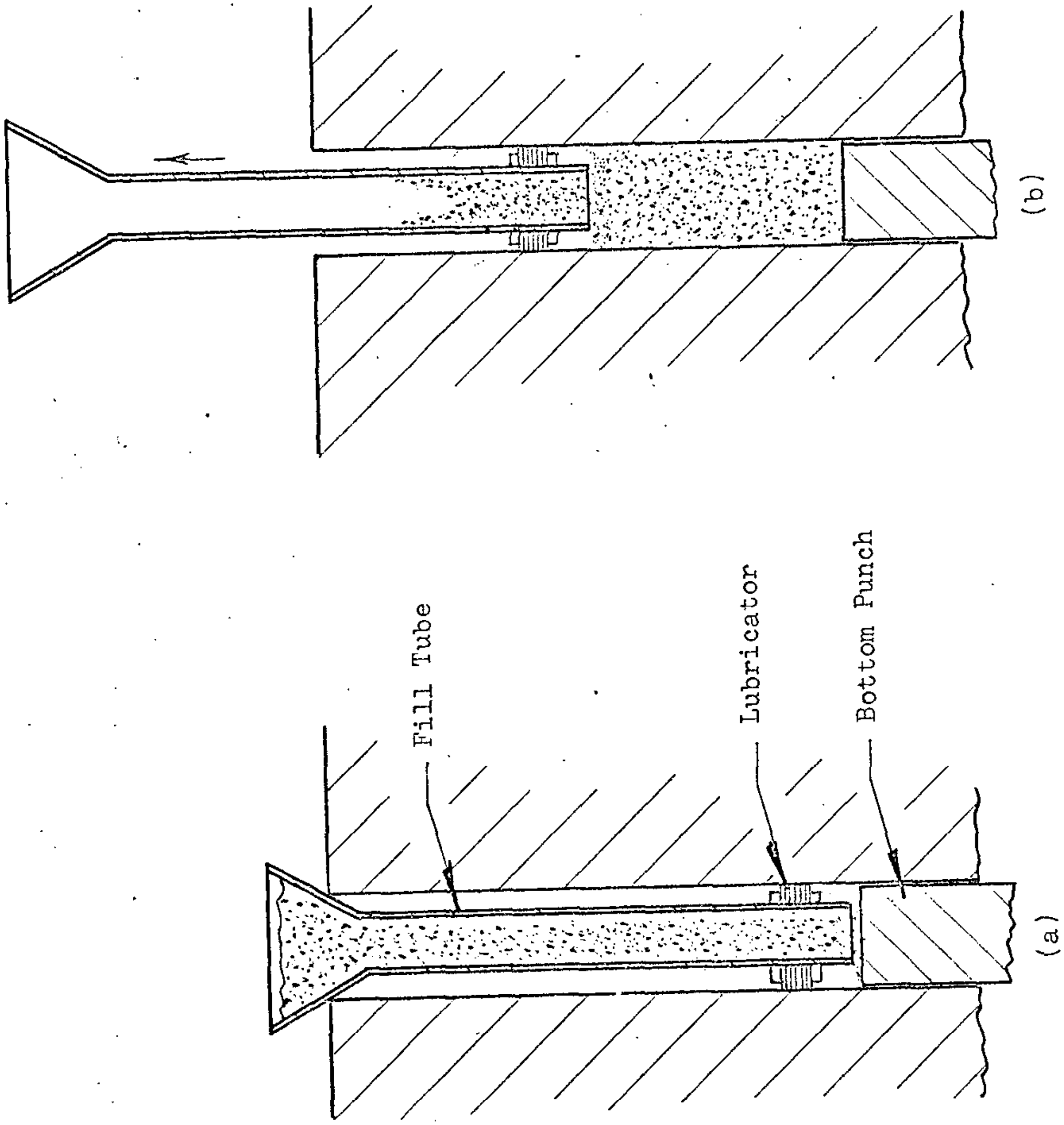

FIGURE 4. TECHNIQUE FOR FILLING AND LUBRICATING COMPACTION DIE.

APPENDIX II

HIGH TEMPERATURE BILLET LEAK TESTING

Leaks too small to be detected by the standard bubble test and leaks that occur when coextrusion billets are heated during processing are detected with a mass spectrometer leak detector attached to a specially designed vacuum chamber, Figure 5. Unlike the bubble test used as a routine check by both SRP and SRL, the test does not determine the exact location of the leak; location of leaks is described below. Because of the extreme sensitivity of the leak detector large leaks must be located (by the bubble test) and repaired before this test.

The test is performed as follows:

- The billet is heated at the outgassing temperature (normally 525°C) for at least 30 minutes.

- It is placed in the test chamber directly from furnace; the chamber is sealed and leak checked.

- When the chamber is determined to be leak tight the billet is pressurized with helium through its evacuation tube to 28-30 psig. The presence of helium in the vacuum chamber as determined by the leak detector indicates a leak in the billet.

- A leak resulting in an output meter reading of 100% on the X50 scale of the leak detector used by SRL (Consolidated Electrodynamics type 24-120B) is the largest allowable leak. This limit is based on a detector sensitivity that will cause an output meter reading of approximately 23% on the X5 scale when exposed to a standard leak of 2.8 x 10^{-8} atm cc He/sec. This limit was established from experience in the ^{242}Pu target-housing tube program. No attempt has been made to determine its exact size, but extrapolation from standard leaks indicates it is approximately 1.22 x 10^{-6} atm cc He/sec.

Billets having leaks greater than the acceptance limit are repaired. An attempt is made to locate the leak by removing it from the test chamber, still pressurized with He, and checking each weld with a sampling probe attached to the detector. Another method is to re-examine the billet in the bubble test tank. If the leak is not located by either of these methods, it is rewelded completely.

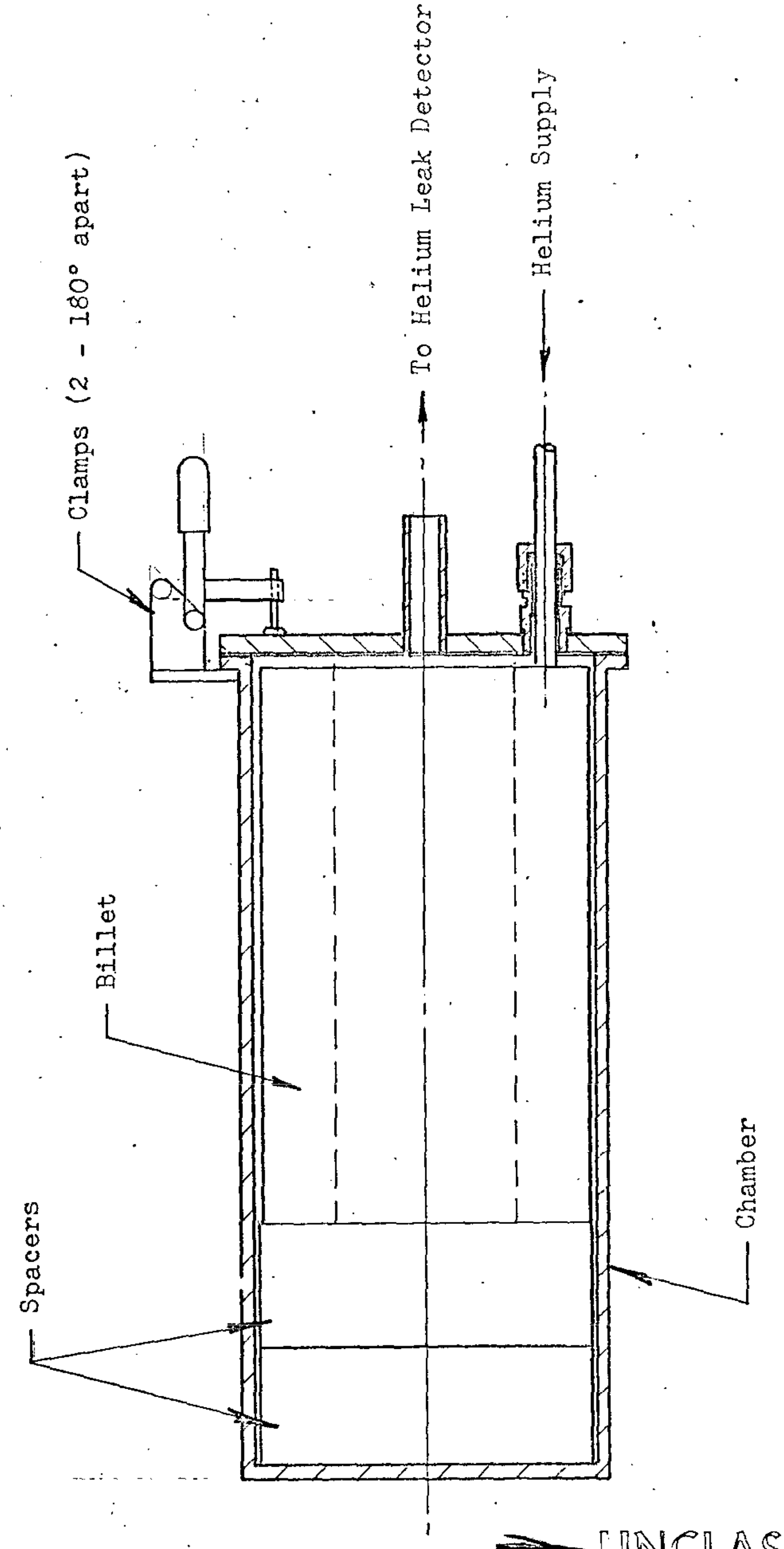

FIGURE 5. HIGH TEMPERATURE HELIUM LEAK TESTING CHAMBER (Ref. Dwg. - ST-PM5-905 and ST-PM5-906)