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R. N. Johnson
Metal Fabrication Development Operation
Metallurgy Development Operation
Hanford Laboratories

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#### FABRICATION OF TARGET ELEMENTS FOR PCTR

R. N. Johnson
Metal Fabrication Development Operation
Hanford Laboratories

### Introduction

A request for fabrication of a load of lithium fluoride-containing target elements for the Physical Constants Test Reactor (PCTR) was received by Metal Fabrication Development Operation. Materials were procured, fabrication methods were developed, and the elements were completed and delivered within a five-week period, meeting the required completion date. Tight specifications on Li<sup>6</sup> content were not only met, but exceeded in all cases. The purpose of this report is two-fold: first, it will provide a complete characterization of each element fabricated, including all pertinent weights and dimensions; and second, it will provide a record of the fabrication methods developed and the details necessary for any future fabrications of similar elements.

### Original Guideline Specifications

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The original guideline specifications stated that the core material was to be aluminum containing 3 wt.% lithium and that the lithium was to be enriched to 41 wt.%  $\text{Li}^6$ . The  $\text{Li}^6$  content had to be known to  $\pm 2.0\%$  for six pieces and  $\pm 3.0\%$  for the remainder of the load. The variation in  $\text{Li}^6$  content was to be within  $\pm 4.0\%$  over the length of the target. The only other specifications given were the external length and diameter of the target element and the cladding materials and thicknesses.

### Core Material

Alloying aluminum and lithium to tight compositional limits has proven to be a very difficult job at best. The wide difference in melting





points between aluminum and lithium, the volatility of lithium, and the reactivity of lithium with crucible materials has forced the metallurgist to rely heavily on chemical and spectrochemical analysis to determine the compositions of his final product. At present the best analyses (to  $\pm$  5%) are not accurate enough to determine the lithium content of the close tolerances required for a PCTR loading.

With the aluminum-lithium alloy core ruled out, lithium compounds were investigated for a possible substitute core material. Of the few lithium compounds readily available, lithium fluoride was the only one that was stable and non-hygroscopic, that contained a high enough lithium content, and that was available in reagent purity as an "off-the-shelf" item. Calculations showed that natural (unenriched) lithium fluoride could provide the same amount of Li<sup>6</sup> per foot of element as that required from the specified Al-Li core. Physics calculations showed that the substitution would be acceptable from the point of view of the information required from the elements in the PCTR.

Table I shows the calculated theoretical densities of LiF required to achieve the same amount of Li<sup>6</sup> per foot of element as the Al-Li alloy core, with the lithium being enriched to several levels of Li<sup>6</sup> content. A lithium fluoride core diameter of 0.616 inch and a coextruded alloy core diameter of 0.629 inch were assumed for the calculations.

### TABLE I.

## Simulated Alloy (Al - 3 wt. % Li)

41% enriched Lithium

50% enriched Lithium

57.3% enriched Lithium

### Density of LiF Required

71.6% TD

87.3% TD

100 % TD



Several methods of fabrication presented themselves, such as: (1) casting rods from molten LiF, (2) filling oversize aluminum tubes with LiF powder and compacting the core by swaging the tube to size, (3) vibrational compaction of a powder core, or (4) pressing the LiF powder into pellets to the required density. The choice was influenced by the fact that very little time was available for the development of fabrication methods.

If feasible, the method of casting rods from molten LiF would have provided a core of essentially 100% theoretical density. The method was acceptable because later communications indicated an interest in higher Li<sup>6</sup> contents than originally specified. A few casting experiments were conducted of LiF using graphite crucibles and molds. The LiF was found to undergo a tremendous volume change on going from liquid to solid, such that piping was a severe problem and several hot toppings were required to achieve even a short section of relatively sound rod. The resulting rod was cracked and had a rough surface, and would have required centerless grinding to achieve an acceptable product. Casting of LiF was abandoned.

Vibrational compaction was eliminated because the available powder was much too fine for this process and the development of the method would have taken too much time.

Swaging a powder filled tube to size was ruled out because of the possibility of density gradients existing in the final product and the difficulty in determining to what extent, if any, these gradients existed. However, with less stringent requirements on density gradients, or with an adequate non-destructive method of determining the density gradients, the swaging process could be an economical method of producing lithium fluoride target elements.





Pelletizing was finally chosen because the lithium fluoride powder could be pressed into pellets of easily determined density which could be varied to duplicate the required Li<sup>6</sup> content by adjusting the pelletizing pressure.

A die was made for the pressing of the pellets. After several modifications the optimum die design was found to consist of the following characteristics.

- (1) The throat of the die should be chrome-plated to reduce friction on the pellet as much as possible. Excessive friction in the die can cause the pellets to crack into laminated layers during removal from the die.
- (2) The optimum clearance between the punches and the walls of the die appears to be between .001 and .002 inch on the diameters. At less than .001 inch clearance, the punches can "freeze" in the die due to the fine powder getting between the punch and the die walls.
- (3) The pellet should travel as short a distance as possible in the die during extraction.
- (4) The die should be "double-acting".

Pellets were pressed at pressures ranging from 40,000 psi to 120,000 psi. Cracking of the pellets was a problem at all but the lowest pressures. By adding 2 wt.% sodium stearate as a binder to the powder, pellets with good green strength and adequate density could be produced at pressures up to 70,000 psi. Above 70,000 psi, cracking again became a problem.

Sintering experiments were conducted in an attempt to further increase the density and the pellet strength, and to remove the sodium stearate binder. Sintering was conducted at temperatures ranging from 250 C to 800 C and for times of 1 hour to 3 hours. The uniformity of the sintered pellets was





inadequate for the PCTR specifications and would have required a fairly high reject rate on the pellets. The sintering cycles required a precise control over heating rate, cooling rate, time, and temperature to obtain an acceptable product. Sintering was discontinued because adequate green strength and uniform densities were obtainable without sintering by pressing the powder at 70,000 psi.

The final pellet manufacturing was as follows:

Manufacturer's Purity Analysis of LiF Powder

Reagent grade lithium fluoride powder was mixed with 2 wt.% sodium stearate by blending in a twin-shell blender for a minimum of eight hours. Steel balls were added to the charge to break up the packing tendency of the powder. The powder was pressed at 70,000 psi in a 0.6155 inch diameter die. The resulting pellets were individually weighed and measured to determine their density. Table II shows the manufacturer's chemical analysis of the impurities in the LiF.

### TABLE II.

Acidity (as HF)	0.002%
Chloride (Cl)	0.005%
Carbonate (Co <sub>3</sub> )	0.007%
Sulfate (SO <sub>4</sub> )	0.005%
Barium (Ba)	0.003%
Heavy Metals (as Pb)	0.004%
Iron (Fe)	0.003%
Potassium (k)	0.008%
Sodium (Na)	0.17 %





### Fabrication of Target Elements

Figure 1 shows a drawing of the PCTR target element. All elements except four were made to the dimensions shown in the drawing. Of the four exceptions, two were made to an exterior length of  $5.379 \pm .015$  inches and two were made to an exterior length of  $23.900 \pm .010$  inches. Figure 2 shows a set of finished elements.

cleaned, weighed, and measured. An aluminum plug (C64 alloy, 5/16 inch long) was TIG welded into one end. The pellets were placed in the aluminum tubes and the ends were closed by TIG welding in another aluminum plug. Gas evolution during welding of the filled tubes caused some problems. Electron beam welding in a vacuum was attempted but the vacuum could not be kept at a low enough pressure. Finally, the problem was solved by drilling a hole in the middle of the cap to allow the evolved gases to escape, TIG welding the cap, then spot welding the hole closed and the closure was completed. The welded ends of the tubes were then swaged down slightly to allow the aluminum tubes to be slipped into the Zircaloy-2 tubes.

The Zircaloy-2 tubing was drawn to final size, straightened, cut to length, cleaned, etched, weighed, and measured. Supports were welded onto the tubing, and iron shoes were crimped onto the supports. The finished aluminum elements were slipped into the Zircaloy-2 tubes, using flake graphite as a lubricant when necessary. (The aluminum had been previously given a light dry blasting to give a surface to which the graphite could stick.) Zircaloy-2 end caps were TIG welded into the ends of the tubes in a helium-filled dry box. The finished elements were then helium leak checked and inspected for final delivery. Figure 3 shows the target element components before assembly.



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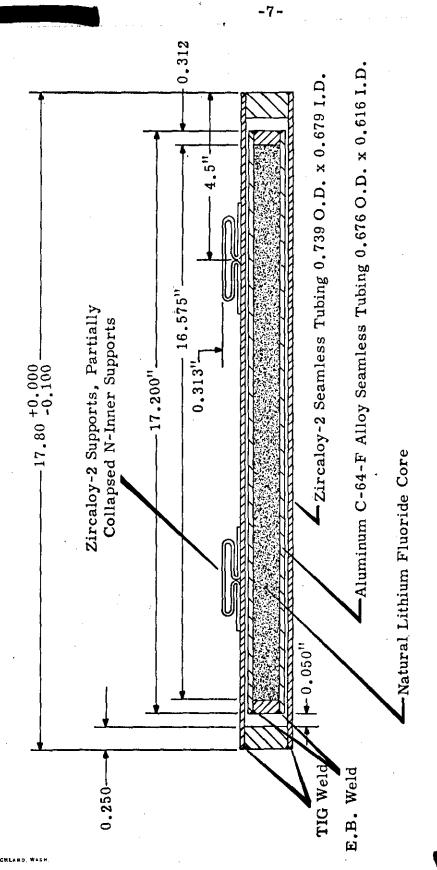


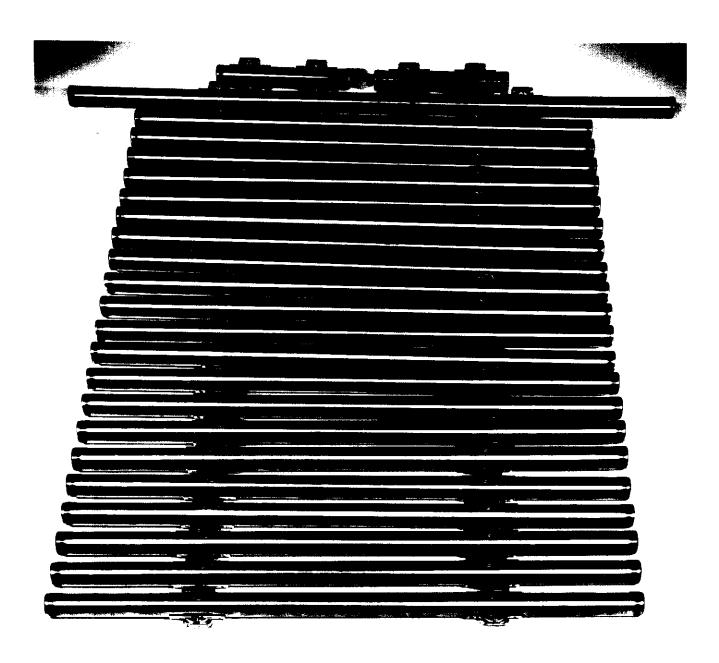
FIGURE 1
Target Element for PCTR

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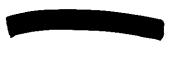
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## FIGURE 2 Finished Target Elements for PCTR





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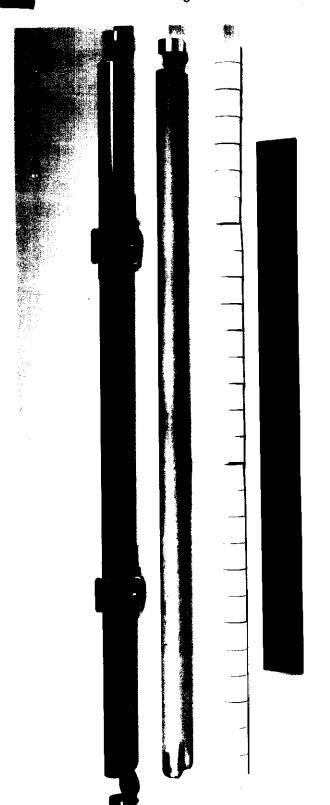
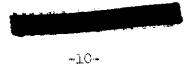


FIGURE 3

PCTR Target Element Components before Assembly



### Detailed Description of Target Elements

The target elements are described by the following tables.

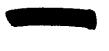
### TABLE III.

### Core Data for 18-inch Target Elements

Element Number	Total Weight of Pellets	Total Weight of LiF	Density of Pellets
1	182.241 gm	178.597 gm	2.252 <u>+</u> 1%
2	185.354 gm	181.647 gm	2.268 <u>+</u> 2%
3	185.349 gm	181.642 gm	2.285 <u>+</u> 1%
8	184.061 gm	180.379 gm	2.260 <u>+</u> 0.5%
9	182.198 gm	178.554 gm	2.259 <u>+</u> 1.5%
10	182.092 gm	178.450 gm	2.264 <u>+</u> 2%
14	184.354 gm	180.667 gm	2.278 <u>+</u> 1.5%
19	185.681 gm	181.968 gm	2.283 <u>+</u> 1%
20	181.563 gm	177.932 gm	2.240 <u>+</u> 1%
21	185.668 gm	181.955 gm	2.292 <u>+</u> 1%
24	186.123 gm	182.401 gm	2.292 <u>+</u> 1%
27	181.893 gm	178.256 gm	2.268 <u>+</u> 2%
28	182.059 gm	178.418 gm	2.254 <u>+</u> 1%
30	182.509 gm	178.859 gm	2.246 <u>+</u> 1%
3:2	185.589 gm	181.877 gm	2.287 <u>+</u> 1%
35	185.824 gm	182.107 gm	2.302 ± 0.4%
36	184.481 gm	180.791 gm	2.282 <u>+</u> 1%
37	185.328 gm	181.622 gm	2.288 <u>+</u> 0.5%
39	185.228 gm	181.524 gm	2.288 <u>+</u> 1%

For all 18-inch elements, total weight of LiF per element is 180.167 gm  $\pm$  1.24%. Average density of the pellets is 2.273 gm/cc, including binder. The average effective density of the LiF is 2.228 gm/cc or 85.66% theoretical density,





This density corresponds to an aluminum - 3 wt.% lithium alloy with the lithium enriched to approximately 49 wt.% Li<sup>6</sup>, assuming the coextruded alloy core had a diameter of 0.629 inch while the pellets were 0.6165 inch diameter. The total Li<sup>6</sup> per element is 3.098 gms ± 1.24%. The 18-inch elements contained an average of 52.4 gms of aluminum each, and an average of 161.9 gms of Zircaloy-2, including supports (of which 2.6 gms is actually iron from the shoes on the supports).

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#### TABLE IV.

### Data on 5.4 in Element

	Element #40	Element #43
Total Weight of Pellets	45.784 gm	45.993 gm
Total Weight of LiF	44.868 gm	45.073 gm
Density of Pellets	2.306 ± 0.4%	2.303 ± 0.2%
Total Li <sup>6</sup> in Element	0.772 gm	0.775 gm
Weight of Al in Element	19.8 gm	19.8 gm
Weight of Zr-2 in Element	75.6 gm	89.0 gm

The 24-inch element was provided for special measurements in the PCTR and its description is more detailed. Table V gives the necessary data on the 24-inch element.

### TABLE V.

### Description of Special 24-inch Element

	Element #44
Total Weight of Pellets	252.021 gm
Total Weight of LiF	246.980 gm
Density of Pellets	2.279 ± 0.4%
Effective Density of LiF	2.233 gm/cc
% Theoretical Density of LiF	85.85% TD
Total Li <sup>6</sup> in Element	4.247 gm



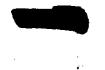


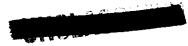
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### TABLE V. (Continued)

	Element #44
Weight of Al Tube	60.1 gm
Weight of Al End Caps	8.3 gm
Weight of Zr Tube	163.9 gm
Weight of Zr End Caps	19.6 gm
Weight of Zr Supports	19.8 gm
Weight of Iron Shoes	2.6 gm





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