CONTROL OF BERYLLIUM-7

IN

LIQUID LITHIUM

R. P. Anantatmula, W. F. Brehm, D. L. Baldwin and J. L. Bevan December 1978

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CONTROL OF BERYLLIUM-7 IN LIQUID LITHIUM

R. P. Anantatmula, W. F. Brehm, D. L. Baldwin and J. L. Bevan Hanford Engineering Development Laboratory

ABSTRACT

Radiation fields created by the production of ⁷Be in lithium of the Fusion Materials Irradiation Test (FMIT) Facility can be sufficiently high to prevent contact maintenance of system components. Preliminary experiments have shown that ⁷Be will adhere strongly to the FMIT piping and components and a good control method for ⁷Be must be developed. The initial experiments have been conducted in static stainless steel capsules and a Modified Thermal Convection Loop (MTCL). The average lithium film thickness on stainless steel was found to be ll um in the temperature range 495°-571°K from the capsule experiments. The short-term experiments showed that ⁷Be migrates to the capsule wall and the long-term experiment indicated that ⁷Be actually penetrates into the stainless steel wall. The diffusion coefficient for ⁷Be in stainless steel at 543°K was calculated to be 5.31 x 10^{-15} cm²/sec. The cold leg of the MTCL picked up much of the ⁷Be activity released into the loop. The diffusion trap, located in the cold leg of the MTCL, was ineffective in removing ⁷Be from lithium, at the very slow flow rates ($< 3.79 \times 10^{-4} \text{ m}^3/\text{s}$) used in the MTCL. Pure iron has been shown to be superior to cobalt and nickel as a getter material for 7Be.

INTRODUCTION

Beryllium-7 will be produced in the lithium target of the FMIT facility by the interaction of the deuteron beam with lithium by the two reactions $^7\text{Li}(d,2n)^7\text{Be}$ and $^6\text{Li}(d,n)^7\text{Be}$. This nuclide has a half-life of 53 days and emits a 0.48 MeV gamma radiation. Previous estimates(1) have shown that an equilibrium concentration of 2.59-x 10^{15} Bq (/0,000 curies) of ^7Be will be present in FMIT lithium.—This is equivalent to a concentration of approximately 1.11 x 10^9 Bq/gm of FMIT lithium and a chemical concentration of 0.25 ppm. Concentrations of ^7Be of this magnitude can lead to radiation fields of over 5.16 x 10^{-3} coulomb/kg.hr(20 R/hr) near FMIT piping and components, more than two orders of magnitude too high for easy contact repair

and maintenance. A recent upper limit calculation of equilibrium concentration of 7 Be in FMIT lithium yielded 2.48 x 10^{16} Bq, which is approximately ten times the previous estimate. Therefore, the need to understand the transport of 7 Be in lithium and to eventually control the spread of the nuclide in the FMIT system has been realized.

A development program has been started recently to identify ⁷Be transport and adherence to metal surfaces in the FMIT temperature range. Also incorporated in the program are studies to remove ⁷Be-from lithium by cold trapping, coprecipitation with non-radioactive beryllium, and selective deposition on materials (trapping).

The present paper describes the results obtained from ⁷Be adherence tests from static capsule studies, and preliminary results on ⁷Be transport in lithium from MTCL experiments.

EXPERIMENTAL PROCEDURE

The lithium needed for the experiments was transferred from a 0.21 m³ lithium drum to a 0.0038 m³ stainless steel tank through a nickel filter. The first set of capsule experiments was performed with lithium in stainless steel capsules, without any ⁷Be, to determine the lithium film thickness on stainless steel as a function of temperature in the FMIT temperature range. For the second series of capsule experiments, the molten lithium in the static capsules was charged with ⁷Be to study the ⁷Be adherence to stainless steel walls and transport in static lithium. The MTCL experiments were designed to understand the ⁷Be transport in slowly flowing lithium and to analyze the effectiveness of the diffusion trap and deposition samples to remove ⁷Be from lithium.

a) <u>Capsule Experiments</u>

The capsules were Type 304 stainless steel, 0.019 m(0.75") 0.D. and 0.017 m(0.68") I.D.. Each capsule was approximately 0.42 m(16.5") long and fitted with a Swagelok* union at one end and the other end welded shut. The capsules were cleaned internally by polishing with emery paper before welding, and cleaned with trichloroethane, acetone and ethanol subsequent to the welding operation. As shown in Figure 1, the capsules were heated

in a quartz tube wrapped with a heater element. The temperature of the capsules was controlled to within ±2°K over the entire length.

Molten lithium at 523°K was drained into the capsules preheated to 523°K from the stainless steel reservoir inside an argon glove box. The glove box was flushed with dry argon 5 or 6 times to remove residual nitrogen and reduce the humidity, prior to filling the capsules with lithium. An analysis of the glove box atmosphere indicated no nitrogen. The lithium in the capsule appeared bright, without any discoloration, indicating no reaction with the glove box atmosphere.

⁷Be for the experiments was obtained from Oak Ridge National Laboratory (ORNL). Using a standard procedure recommended by ORNL, the ⁷Be needed for the capsule experiments was converted to its nitrate. An aliquot of the salt was pipetted onto a series of stainless steel planchettes and dried. The nitrate was then ignited at 873°K to form the oxide. Each planchette was then bottled individually and counted by gamma spectroscopic analysis (GSA). A single planchette was then wrapped with a stainless steel wire to the center of the thermocouple tube and inserted into the capsule.

At the end of each run, the capsule was drained in the glove box into a small stainless steel reservoir. The planchette and the drained lithium were counted for ⁷Be activity by GSA. The capsule was then sectioned into several pieces and the lithium film from each piece dissolved in methanol/water baths and the solution and the capsule piece counted for ⁷Be activity by GSA. Aliquots of solution were subsequently_analyzed_for lithium by Atomic_Absorption Spectroscopy.

b) MTCL Experiments

The MTCL is schematically illustrated in Figure 2. The vertical and horizontal sections of the cold leg and hot leg are approximately 0.41 m(16") and 0.36 m(14") respectively. Tackwelded stainless steel pins were used to secure the deposition samples to the hot and cold leg thermocouple tubes. The samples were of iron (99.64%), cobalt (99.88%) and nickel (99.98%), 0.025 m x 0.01 m, with thicknesses ranging from 1.27 x 10^{-4} m for iron to

2.54 x 10^{-4} m for nickel to 6.35 x 10^{-4} m for cobalt. A diffusion cold trap was provided at the bottom of the cold leg. Due to the high surface tension and low density of lithium at FMIT operating temperatures, a linear induction pump (< 3.79 x 10^{-4} m³/s) was used to aid the thermal convection flow, hence the name MTCL. After initially heating the MTCL to a uniform temperature above 543°K (270°C), a temperature gradient of 54°K between the hot leg (543°K) and the cold leg (489°K), close to that of the FMIT temperature gradient, was maintained by providing a constant heat input to the hot leg and cooling the cold leg by blowers. The pump had to be operated at a very low power input to maintain the temperature gradient.

Lithium flow at a very small rate was established from the hot leg through the pump to the cold leg.

For the MTCL experiments, ⁷Be embedded in the lithium target material (probably in nitride form) was introduced in the upper part of the hot leg in the stagnant lithium. The loop was filled with lithium from the reservoir by evacuating the MTCL and applying 3.4 x 10⁴ pascals (5 psi) argon pressure on the reservoir. While the MTCL was operating, gamma counting of the loop was performed by feeding the count output from a NaI detector into a computerized multichannel analyzer. At the completion of the run, lithium from the MTCL was drained under argon pressure, and the drained lithium was gamma-counted by GSA, similar to the capsule experiments. A Germanium-Lithium detector was used in conjunction with the multichannel analyzer to analyze the ⁷Be activity remaining on the deposition samples and the MTCL piping.

RESULTS AND DISCUSSION

a) <u>Capsule Experiments</u>

As mentioned earlier, the first set of experiments was aimed at determining the lithium film thickness on drained stainless steel surfaces. Four runs were made for this set of experiments. The capsules were maintained at 515°, 537°, 495° and 571°K respectively for approximately 24 hours. At the end of each run, the straight section of each capsule was cut into three pieces and each piece analyzed for lithium concentration along the length of the tube. The results from the four runs are reported in Table 1.

As can be seen from the table, the lithium film thickness ranges from 8.4 µm to 12.3 µm in the temperature range 495°-571°K. Cowles and Pasternak(2) report that pure lithium will not wet stainless steel until 673°K, and the wetting temperature for impure lithium is 755°K. However, the phenomenon of wetting is not only a function of temperature, but also a function of time at a given temperature, where the contact angle between the liquid and the solid, in general, decreases with increasing time(3). Furthermore, the wetting temperature is raised by the presence of exide film on a metal surface. The stainless steel capsules used in the present investigations were cleaned thoroughly to minimize the wetting problem from the exide film. In our experiments we have not detected any globules of lithium sticking to the straight surface of the capsule to introduce an error in the film thicknes calculation. However, we have made the assumption that the lithium film was distributed uniformly over the entire surface area of the capsule section examined.

The average film thickness from all four runs was 11.0 µm in the temperature range 495°-571°K. Because of its high surface tension and low density, lithium could not be poured from one capsule to another at 463°K, precluding film thickness measurement at that temperature. At all the remaining temperatures of investigation, the top capsule had to be tapped to start the lithium flow into the bottom capsule, again due to the low density and high surface tension of lithium.

The second set of experiments was performed with ⁷Be in oxide form. As before, four runs were made for these experiments. For Run 5, the planchette containing 4.03 x 10⁷ Bq (1.09 mCi) of ⁷Be was heated in the capsule containing lithium to 543°K and kept overnight for a period of 17 hours. Then the capsule was cut into 9 pieces and analyzed for ⁷Be and lithium. As reported in Table 2, 91% of the ⁷Be activity remained on the planchette, while 3% was retained in the lithium film and approximately 6% was present in the drained lithium. The ⁷Be activity in the lithium film was 718 Bq/mg as compared with 44 Bq/mg of drained lithium. This is a factor of 16 higher for the lithium film, which indicated that ⁷Be is segregating at capsule walls. Furthermore, the ⁷Be activity increased with increasing lithium film from bottom to top of the capsule. The average lithium film thickness on the straight

section of the capsule was computed to be 15.7 μ m, which is slightly higher than the \sim 12 μ m obtained at 543°K in the first set of experiments.

Since the majority of ⁷Be activity was retained on the planchette in Run 5, an alternate procedure was used for Run 6. The lithium was heated to 693°K for 3 hours and then maintained at 543°K for 17 hours. A Geiger-Mueller counter indicated that much of the activity was still in the vicinity of the planchette. Therefore, the capsule was heated to 838°K and kept there for 5 hours and then maintained at 543°K for approximately 17 hours in an attempt to release most of the ⁷Be from the planchette. Lithium was then drained from the capsule and lithium and ⁷Be analysis performed.

The results of Run 6 are described in Table 3. It may be noted from Table 3 that our attempt to release majority of ^7Be activity into lithium was successful. The drained lithium contained 69% of the original activity on the planchette. The ^7Be activity in the lithium film was 25%, while the activity remaining on the planchette was only 6%. As for Run 5, the ^7Be concentration of lithium in the lithium film was a factor of \sim 16 higher than that in the drained lithium, supporting our earlier conclusion that ^7Be may be migrating toward the capsule walls. An estimate of the lithium film thickness on the stainless steel yielded a value of 14 μm at 543°K, which compares favorably with the data presented above.

Test Run 7 was performed at 523°K. The capsule was heated to 798°K and kept at that temperature for 45 minutes prior to equilibrating at 523°K for 17 hours. As pointed out earlier, the high temperature excursion is mainly to release as much 7Be into lithium as possible, in order to get reliable 7Be adherence data. Gamma-count data indicated that 16% of the original 7Be activity remained on the planchette, with 41% of the activity present in drained lithium, 43% in the lithium adhering to the Swagelok fitting and the capsule body. The 7Be concentration of lithium retained on the capsule was a factor of 71 higher than that in the drained lithium; again substantiating our conclusion that 7Be may be migrating toward the capsule walls. The lithium film thickness on the straight section of the capsule (excluding the Swagelok fitting and the capsule bottom) was computed as 8.5 µm at 523°K, in excellent agreement with the 8.4 µm reported in Table 1 at 515°K.

It was not readily apparent from the data presented above if $^7\mathrm{Be}$ actually diffuses into the austenitic stainless steel.

The ⁷Be was mainly contained in the lithium film and was easily washed away with the lithium film, leaving no traces of activity on the walls. To investigate the phenomenon of ⁷Be diffusion more completely, we have performed Run 8 for 4000 hours at 543°K.

The results of Run 8 are tabulated in Table 4. 7 Be could not be removed by methanol/water rinses for segments 6 through 9. The segments were dissolved in acid (HCL + HNO₃) baths to remove 7 Be completely. This implies that 7 Be actually penetrated into the stainless steel wall. The thickness of stainless steel surface layer removed by the acid was estimated for the segments (Table 6) by analyzing the solutions for chromium and extrapolating to obtain the mass of stainless steel removed.

The diffusion coefficient (D) of 7Be in austenitic stainless steel was calculated to be 5.31 x 10^{-15} cm 2 /sec at 543°K from the depth of penetration into segment 6, assuming D to be independent of composition of steel. We have chosen segment 6 due to its proximity to the 7Be source; it is therefore safe to assume that we have a constant source of 7Be from the start of the 4000-hour experiment. The D value of 7Be calculated here is much higher than the value of 6.53×10^{-25} cm 2 /sec computed from the diffusion equation derived by Grigoryev and Pavlinov (4) for D of 7Be in γ -iron. The difference in the D values may be explained in the following manner. The equation of reference 4 was derived from the data in the temperature range $1373^\circ - 1623^\circ K$, and at high temperatures lattice diffusion is important. However, at lower temperatures, diffusion is mainly through the grain boundaries and grain boundary diffusion is several orders of magnitude higher than lattice diffusion. In addition, the diffusion of 7Be is expected to be affected by the presence of other alloying elements in steel.

The lithium film thickness for Run 8 was estimated as 10.2 μm at 543%K, in good agreement with the data presented here.

(b) MTCL Experiment

Only one run (Run 1, MTCL) was performed with the MTCL at 543°K hot leg and 489°K cold leg. The preliminary results are reported here in the following. The data analysis, however, is still continuing.

As shown in Figure 3, the cold leg vertical section has picked up half.... the final equilibrium ⁷Be activity within the first 30 hours of MTCL operation. After approximately 300 hours of operation, the 7Be activity in the cold leg reached a plateau, indicating that the rate of pickup is equal to the rate of decay. The diffusion trap, on the other hand, has reached its plateau of ⁷Be activity after approximately the first 100 hours of operation. The final equilibrium activity in the diffusion trap is less than half the activity found in the cold leg. Very little activity was noted in the hot leg region, in both the horizontal and the vertical sections. However, buildup of ⁷Be activity was observed at the 90° elbow between the hot leg and the pump at levels much higher than in the rest of the hot leg piping. A maximum in ⁷Be activity was seen in the cold leg about 0.10 m(4") above the bottom tee (Figure 4). This is the same place where a minimum in temperature was also recorded. During the operation of the loop, ⁷Be activity under the pump could not be measured due to the interference of the magnetic field generated by the pump coils with the operation of the photomultiplier of the detector.

The MTCL was shut down after 975 hours of operation and lithium drained. Gamma counting of the loop piping, the drained lithium and the deposition samples was performed. Accounting-for-decay,-about-93% of the ⁷Be activity was present on the piping, 6% in the drained lithium and 1% on the deposition samples. Approximately 50% of the ⁷Be activity has been accounted for in the vicinity of the ⁷Be source in the dead-leg piping of the hot leg. Among the deposition samples, iron has picked up the most activity. This is not too surprising since the solubility of beryllium (5) is greater in iron at FMIT temperatures than solutility in cobalt and nickel. The gamma-counting of the samples was made after removing the lithium film, while the MTCL piping was counted with lithium film in place. Therefore, on the basis of the capsule experiments, one can say that much of the 93% of the activity detected on the MTCL piping is quite possibly contained in the lithium film. The lithium film analysis was not carried out for the MTCL piping due to the different temperatures and 7Be activities in different parts of the loop.

It is quite possible that the deposition samples would have picked up more ⁷Be if more was available in the flowing lithium. Run 2, MTCL is currently in progress to check not only the reproducibility of the preliminary data presented here, but also find out if more ⁷Be could be deposited onto the samples. The ⁷Be source for the second run is located in flowing lithium in the hot leg, upstream from the deposition samples.

CONCLUSIONS

It can be concluded from the present investigations that ⁷Be adheres to the steel wall in contact after prolonged exposure time at FMIT temperatures, and has a tendency to segregate to the cold leg of the loop. The diffusion trap has been shown to be ineffective under conditions of very small flow rates. Very little ⁷Be activity can be removed by draining the lithium from the loop after prolonged operation. This means that control methods must be developed to isolate ⁷Be in parts of the loop that can be removed and replaced for easy contact maintenance of FMIT. Experiments are currently underway to investigate the effect of doping the

hot leg lithium with inert beryllium to precipitate $^7\mathrm{Be}$ in the cold leg. Additional experiments with deposition samples are also continuing to verify the reproducibility of the data presented here and to develop a trap material for $^7\mathrm{Be}$.

ACKNOWLEDGMENTS

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TABLE 1. LITHIUM FILM THICKNESS MEASUREMENTS

Run #	 Temperature °K		Average Lithium Film Thickness(um)
1	515		8.4
2	537	. ·	11.1
3	 495		12.2
4	571		12.3

TABLE 2. RESULTS OF RUN 5 (543°K) WITH ⁷Be

Segment	Length(m)	Li(mg)	⁷ Be(Bq)
· ·			
Bottom -	0.025	32.0	3.59 x 10 ⁴
1	0.05	13.5	4.81×10^4
2	0.05	15.5	5.18×10^4
3	0.05	19.0	5.92 x 10 ⁴
4	0.05	21.2	8.14×10^4
5	0.05	24.0	9.99×10^4
6	0.05	26.6	11.84 x 10 ⁴
7	0.05	31.5	13.32×10^4
Swagelok Fitting	0.075	1160.0	33.67 x 10 ⁴
Drained lithium		43.5 gm	192.40 x 10 ⁴
Planchette			35.89 x 10 ⁶

TABLE 3. RESULTS OF RUN 6 (543°K) WITH ⁷Be

Segment	Length (m)	Li (mg)	⁷ Be (Bq)
Bottom	0.025	42.5	5.12 x 10 ⁵
1	0.05	9.8	6.42 x 10 ⁵
2	0.05	11.8	7.25×10^{5}
3	0.05	13.3	1.38×10^6
4	0.05	13.3	1.04 x 10 ⁶
5	0.05	15.5	1.86×10^6
6	0.05	20.5	1.05 x 10 ⁶
. 7	0.05	32.8	1.04×10^{6}
8	0.043	33.8	1.05 x 10 ⁶
Swagelok Fitting	0.033	800	4.42 x 10 ⁶
Drained lithium		43.5 gm	3.84×10^7
Planchette			3.38×10^6

TABLE 4. RESULTS OF 4000-HOUR ⁷Be TEST AT 543°K

Segment	Length(m)	Steel Removed(mg)	Depth of ⁷ Be Penetration (µm)
6	0.051	122	5.53
7	0.051	101	4.58
8	0.051	31	1.41
9	0.038	9	0.54

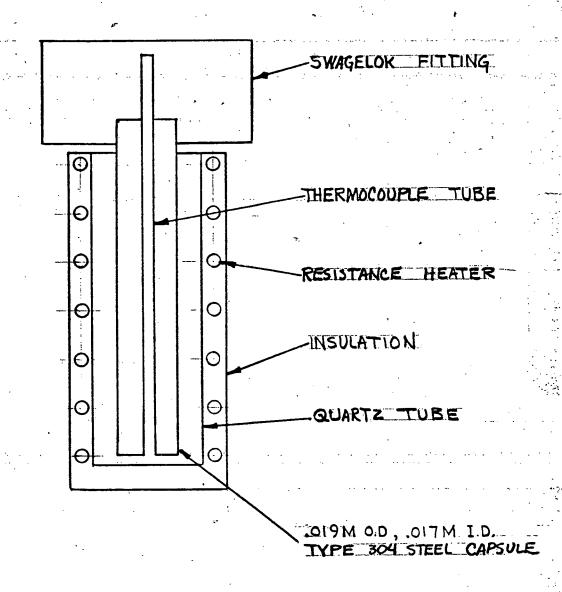


FIGURE 1. SCHEMATIC OF CAPSULE ARRANGEMENT

· THERMOCOUPLE LOCATION

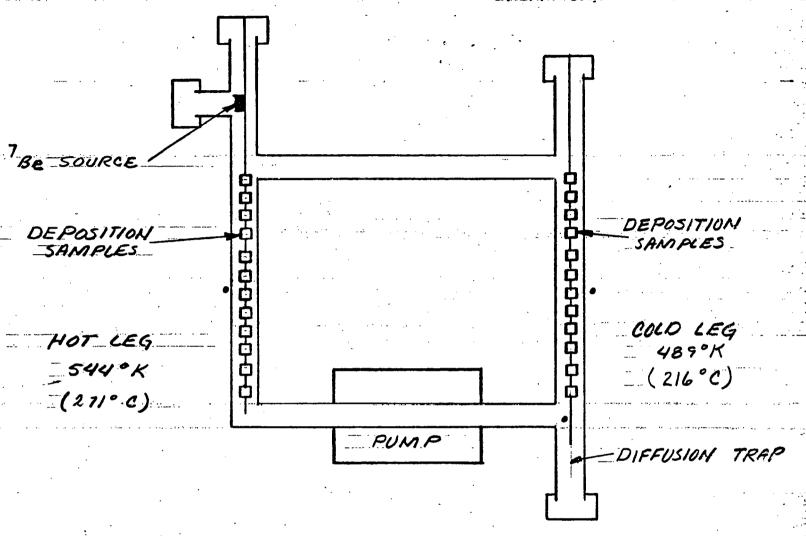


FIGURE 2. MODIFIED THERMAL CONVECTION LOOP SCHEMATIC.

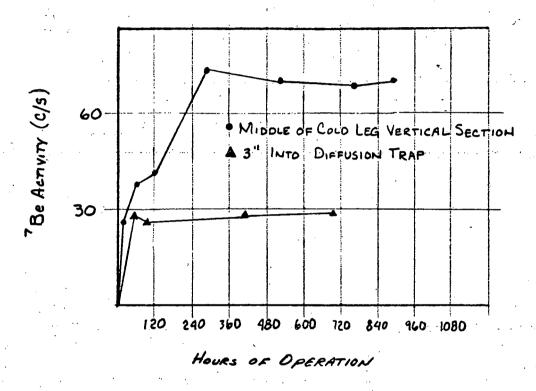


FIGURE 3. $^{7}_{\text{Be}}$ DISTRIBUTION DURING MTCL OPERATION

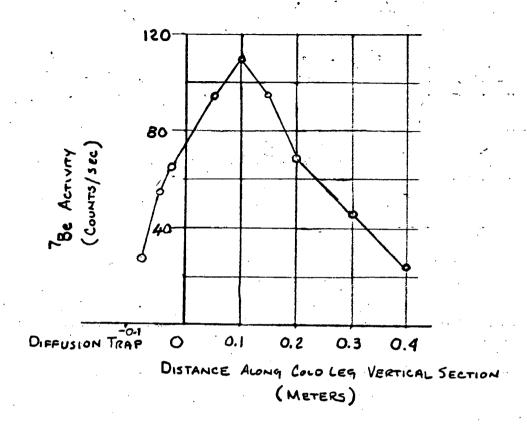
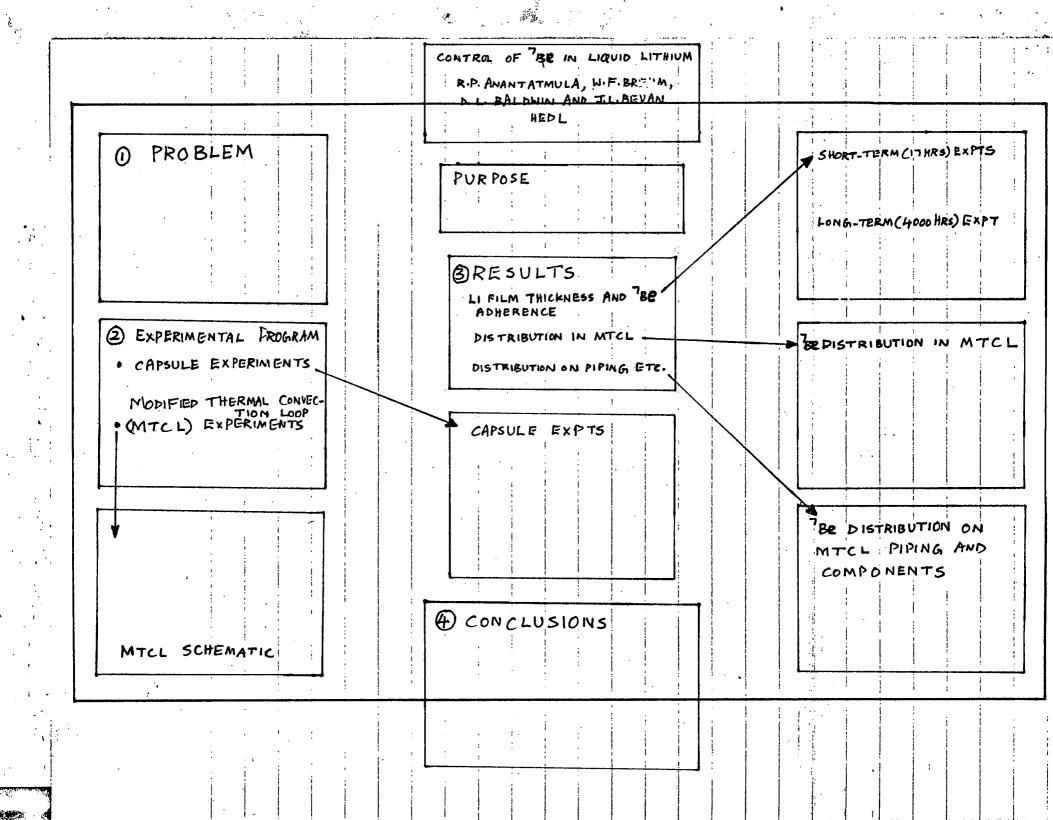


FIGURE 4. ⁷Be DISTRIBUTION IN MTCL COLD LEG AFTER APPROXIMATELY 800 HOURS OF OPERATION - TEMPERATURE (~ 483°K)

MATERIAL TO BE PRESENTED AT THE POSTER SESSION



R. P. ANANTATMULA, W. F. BREHM, D. L. BALDWIN AND J. L. BEVAN

HANFORD ENGINEERING DEVELOPMENT LABORATORY

PURPOSE

. TO DEVELOP A METHOD TO CONTROL THE TRANSPORT OF $^7\mathrm{Be}$ IN FUSION MATERIALS IRRADIATION TEST (FMIT) FACILITY

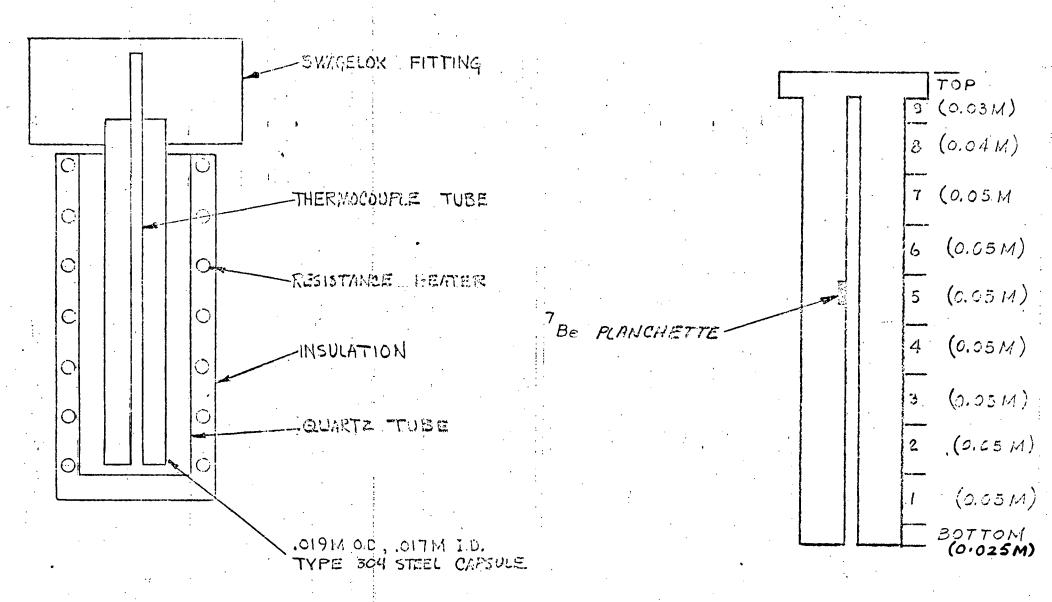
(1) PROBLEM

- ⁷BE WILL BE PRODUCED IN FMIT LITHIUM TARGET BY INTERACTION OF DEUTERON BEAM WITH LITHIUM
- . RECENT ESTIMATES SHOW THAT AN EQUILIBRIUM CONCENTRATION OF AT LEAST 2.59 \times 10 15 BQ of ^7Be WILL BE PRESENT IN FMIT LITHIUM
- . THIS CONCENTRATION OF 7BE LEADS TO RADIATION FIELDS OF OVER 5.16 x 10^{-3} COULOMB/KG.HR (20 R/HR) PREVENTING CONTACT MAINTENANCE OF FMIT PIPING AND COMPONENTS

(2) EXPERIMENTAL PROGRAM

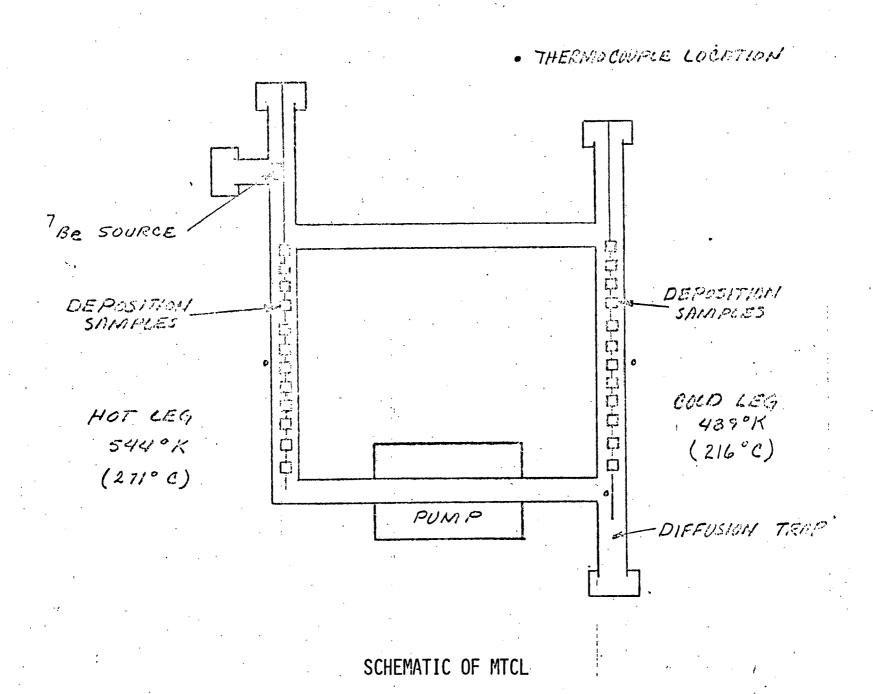
- CAPSULE EXPERIMENTS

 TO DETERMINE LITHIUM FILM THICKNESS ON AND ⁷BE ADHERENCE TO STAINLESS STEEL AT FMIT TEMPERATURE (473°-573°K)
 - MODIFIED THERMAL CONVECTION LOOP (MTCL) EXPERIMENTS (HOT LEG 543°K, COLD LEG 489°K)
 - TO IDENTIFY ⁷BE TRANSPORT IN A TEMPERATURE GRADIENT
 - TO TEST EFFECTIVENESS OF DIFFUSION TRAP IN PICKING UP $^7\mathrm{Be}$ IN SLOWLY FLOWING LITHIUM
- TO DETERMINE THE ABILITY OF FE, Co AND NI SAMPLES
 IN GETTERING ⁷BE



CAPSULE ARRANGEMENT

SECTIONING OF CAPSULE



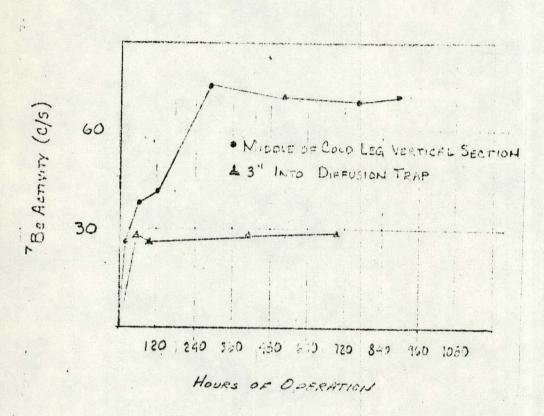
(3) RESULTS

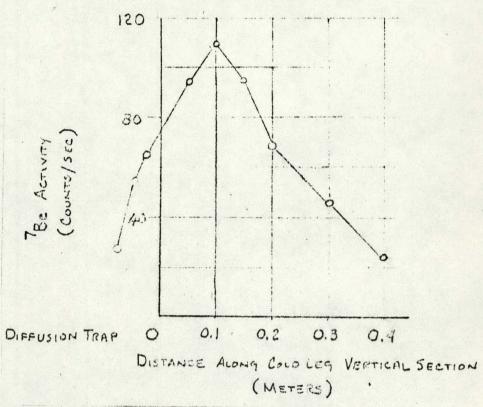
- LITHIUM FILM THICKNESS AND ⁷Be ADHERENCE
- DISTRIBUTION OF ⁷Be IN MTCL DURING OPERATION
 - DISTRIBUTION OF $7_{\mbox{\footnotesize{BE}}}$ ON MTCL PIPING AND COMPONENTS

LITHIUM FILM THICKNESS AND ⁷BE ADHERENCE MEASUREMENTS

- SHORT-TERM EXPERIMENTS (17, 24 HRS)
 - . AVERAGE LITHIUM FILM THICKNESS IS 11 ${\scriptscriptstyle \mu M}$ IN THE TEMPERATURE RANGE $495^{o}{-}571^{o}{\rm K}$
 - ⁷BE PREFERENTIALLY MIGRATES TO CAPSULE WALL
- LONG-TERM EXPERIMENT (4,000 HRS)
 - . 7BE PENETRATES INTO THE STAINLESS STEEL CAPSULE WALL AT FMIT TEMPERATURES
 - . DIFFUSION COEFFICIENT OF $^7\mathrm{Be}$ IN STEEL 5.31 \times 10⁻¹⁵ cm²/sec AT 543°K

7BE DISTRIBUTION IN MTCL (DURING OPERATION)





⁷Be DISTRIBUTION IN MTCL COLD LEG AFTER APPROXIMATELY 800 HOURS OF OPERATION - TEMPERATURE (~ 483°K)

⁷BE DISTRIBUTION ON MTCL PIPING AND COMPONENTS (AFTER 975 HRS OF OPERATION)

- 93% OF 7BE ACTIVITY PRESENT ON PIPING
 - 50% PRESENT IN THE VICINITY OF 7BE SOURCE
- 6% OF ⁷BE ACTIVITY PRESENT IN DRAINED LITHIUM
- 1% OF ⁷BE ACTIVITY PRESENT ON DEPOSITION SAMPLES
 - IRON SAMPLES PICKED UP MORE ACTIVITY COMPARED WITH COBALT AND NICKEL SAMPLES

(4) CONCLUSIONS

- . ⁷BE ADHERES TO STAINLESS STEEL AFTER PROLONGED EXPOSURE AT FMIT TEMPERATURES
- ⁷BE SEGREGATES TO THE COLD LEG OF A LITHIUM LOOP
- . DIFFUSION TRAP IS NOT VERY EFFECTIVE IN REMOVING $^7\mathrm{Be}$ FROM LITHIUM AT LOW FLOW RATES
- VERY LITTLE ⁷Be CAN BE REMOVED BY DRAINING LITHIUM FROM THE LOOP AFTER PROLONGED OPERATION
- . CONTROL METHODS MUST BE DEVELOPED TO ISOLATE $^7\mathrm{Be}$ IN PARTS OF THE LOOP THAT CAN BE REMOVED AND REPLACED