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TO REMOVE TRITIUM FROM LIQUID LITHIUM FUSION REACTOR BLANKETS

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Presented at:

First ANS Topical Meeting on the Technology of Controlled
Nuclear Fusion, San Diego, California, April 16-18, 1974

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*Work performed under the auspices of the U. S. Atomic Energy
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ABSTRACT

A method for removing tritium from liquid lithium fusion reactor blankets by extraction with a molten salt is described. Results of distribution coefficient measurements made with lithium-lithium halide mixtures have demonstrated that tritium is preferentially distributed in the salt phase by a factor greater than 1.0 on a volumetric basis. Other considerations related to (i) mutual solubilities between the salt and metal, (ii) phase separation, (iii) blanket neutronics, (iv) corrosion, (v) fabrication, and (vi) recovery of tritium from the salt phase indicate that the extraction process should be feasible. Calculations based on the blanket processing requirements for the IASL/ANL Reference Theta-Pinch Reactor (RTPR) show that the equipment and power needed to carry out a molten-salt extraction operation on the lithium blanket of the RTPR are reasonable.

INTRODUCTION

Future DT-fueled fusion power plants will require an efficient processing system that recovers tritium from the blanket at a rate equal to the breeding rate and at the same time maintains a low steady-state inventory of tritium throughout the blanket region. The desirability of a minimal tritium inventory in the blanket stems from both economic and safety considerations.¹ If, after start-up of a DT-fueled plant, large amounts of tritium are allowed to build up in the blanket before the recovery rate of the tritium processor can match the breeding rate, it will be necessary to begin operation of the plant with a substantial tritium reserve for fueling. Furthermore, as the tritium concentration in the blanket increases so will the partial pressure of tritium in equilibrium with the blanket. This will result in an increased rate of tritium permeation through the blanket structure and primary heat exchanger. Large quantities of tritium in the

*This work was performed under the auspices of the U. S. Atomic Energy Commission.

blanket system will also complicate maintenance problems, and could increase the hazards associated with mechanical and structural failures, fires and other abnormalities.

For DT-fueled plants with liquid lithium blankets, the requirement of a minimal tritium inventory is complicated by the tremendous capacity of lithium to retain tritium relative to most other materials.¹ Because liquid lithium is a leading candidate for the breeding medium in DT-reactor concepts, the problem of tritium recovery from lithium deserves special attention. It has been shown² that processes based on gas sparging, distillation, evaporation, or mechanically pumped permeable membranes are not likely to reduce the tritium concentration in a lithium blanket to acceptable levels (probably < 10 ppm). Schemes that employ solid, hydrogen-gettering materials have the potential to provide adequate tritium removal, but procedures for the maintenance and regeneration of the gettering substance remain to be demonstrated.

A method developed for fission reactor fuel processing, which could have applications in fusion reactor technology, involves removal of fuel materials from liquid metals by extraction with a molten salt.³ Some prior experimental investigations⁴ conducted in our laboratory on lithium/hydrogen galvanic cells with fused lithium halide electrolytes had indicated that the lithium hydride formed during discharge was preferentially extracted into the electrolyte. More recently a series of studies has been carried out to measure distribution coefficients for tritium between liquid lithium and selected molten lithium halide-containing eutectics. Results of these distribution studies in combination with existing data on other pertinent physical, chemical and electrochemical properties of liquid lithium and molten lithium halides have permitted a preliminary assessment of the feasibility and practicality of using a molten-salt extraction process to remove tritium from liquid lithium fusion reactor blankets.

EXPERIMENTAL RESULTS

Distribution coefficients for tritium between liquid lithium and selected lithium halide-containing molten-salt eutectics were measured in closed-capsule experiments. Samples containing lithium metal and a salt-eutectic were sealed in Nb-1%Zr capsules, heated to 600°C in the presence of an argon carrier gas containing 2 μ Ci of tritium per standard liter (the tritium entering the capsule by permeation), equilibrated at temperatures between 400 and 600°C, and rapidly quenched. The capsules were then opened and portions of both the lithium phase and the salt phase were analyzed for tritium content by standard dissolution and counting techniques.⁵

The results of these studies (summarized in Table I) show that attractive (from a processing viewpoint) distributions of LiT between the salt and metal phases can be achieved with all three eutectics. Taking into account melting point, relative ease of handling, and extraction potential, the LiCl-KCl eutectic appears to be best suited for implementation as the extracting salt. The LiF-LiCl eutectic, although higher melting and more corrosive, could find application in

high-temperature ($\geq 800^\circ\text{C}$) blanket designs. For reasons that are discussed later on, the LiBr-RbBr eutectic appears to be neutronically less favorable and will probably turn out to be considerably more expensive.

It is not surprising that LiT, which is largely a salt-like substance, favors the salt phase. In fact, other salt-like impurities (e.g., Li_2O , Li_2C_2 and Li_3N as well as oxides, carbides, and nitrides of the structural materials) that develop in the lithium circuits, might also be preferentially extracted into the salt phase. However, since data on the partitioning of species other than LiT between liquid lithium and molten lithium halides remain to be collected, the rest of this study will consider only the tritium-recovery application.

OPERATIONAL FEATURES OF THE EXTRACTION SCHEME

The results presented in Table I imply that in a molten-salt extraction scheme in which some fraction of the lithium inventory of a fusion reactor is continuously contacted with an equal volume of salt, the salt phase can remove at least 50% of the tritium present in the incoming lithium on each pass. A plausible flow diagram for such a scheme is given in Fig. 1. The high-temperature lithium coming from the blanket is heat-exchanged with a secondary heat-transfer fluid or a working fluid and passed on to a holding tank prior to recirculation through the blanket region. A portion of the lithium flow coming from the heat exchanger is diverted to the extraction equipment where it is contacted with and separated from a molten salt and then returned to the holding tank. The salt coming from the extraction equipment is circulated to an electrolytic recovery tank where the extracted tritium is removed by electrochemical oxidation of the tritide ion (T^-) to T_2 at a collection-electrode.

To effectively implement this process in currently conceived DT-fueled fusion reactors, a number of conditions (in addition to favorable equilibrium distribution) must be met. The two fluids must be separable once they are mixed and the mutual solubilities will probably have to be negligibly small. The tritium must be recoverable from the salt phase after electrochemical evolution, and chemical effects resulting from the presence of liquid lithium and molten alkali halides in close proximity must not cause excessive corrosion in the contactor equipment or elsewhere in the lithium circuits and in the salt loop. Existing data related to these potential problem areas are summarized briefly below:

Separability: Years of experience in our laboratory with liquid lithium/molten salt systems have shown that molten salts are easily separated from liquid lithium by gravity alone and that emulsification should not present any problems. Lithium is less dense than all three of the salts indicated in Table I by a factor of at least three.⁶⁻⁹ This should result in rapid separation of the liquid lithium/molten salt slurries formed during the mixing step in Fig. 1, even if gravitational settling is the only separation procedure. If centrifugal action is applied to the slurries, separation would be greatly accelerated and minimal physical entrainment of the two phases in one

another should result.

Mutual solubilities: The mutual solubilities of lithium in the salt phase and salt in the lithium phase cannot be overcome by any physical separation method. At the operating temperature of the mixing and separating equipment each phase will undoubtedly reach its saturation solubility in the other. Problems resulting from lithium in the salt phase could probably be nullified by electro-oxidizing the dissolved lithium soon after the salt leaves the separating equipment.

The consequences of having salt dissolved in the lithium can be of greater significance. It does not appear possible to remove other alkali metals or halide species present in the lithium without a cumbersome processing step; hence, they will probably have to be tolerated and their effects on blanket neutronics, corrosion, etc. will require careful investigation. Solubility data¹⁰⁻¹² for lithium in LiCl and in LiCl-KCl and for LiCl in lithium show that at temperatures below 600°C the solubility of lithium in the salts is less than 1 mole % and, more importantly, the solubility of LiCl in lithium is less than 0.02 mole %. The latter value corresponds to less than 500 weight ppm of chloride in the lithium (at 600°C), and at temperatures around 450°C, the chloride level would be about 100 ppm.

Preliminary neutronics calculations¹³ have indicated that a few hundred ppm of ¹⁹F, ³⁹K, ³⁵Cl, or ³⁷Cl in the lithium will have no measurable impact on tritium breeding, induced activity, or long-lived transmutation-product levels. Chlorine (natural isotopic abundance), has a relatively high thermal neutron cross section (about 430 barns) compared to the tritium production cross section of ⁶Li (70 barns), but for a chloride level of 500 ppm (by weight) in lithium (natural isotopic abundance) the macroscopic absorption cross section of the chlorine is only 0.06% of the macroscopic tritium production cross section of the ⁶Li present. This perturbation is well within the present uncertainty in the ⁶Li(n,α)T cross section. The total neutron absorption cross section of ³⁹K is more than two orders of magnitude lower than the chlorine cross section; hence its effect on tritium breeding will be even less significant at concentrations around 500 ppm.

With regard to long-lived radioactive-isotope production resulting from the presence of KCl in the blanket fluid, all possible transmutation products from incident neutrons, photons, alpha-particles, and recoil tritium atoms on ³⁵Cl, ³⁷Cl, and ³⁹K were considered. Only two long-lived isotopes were found.

The β^- emission of these decays is only a negligible fraction of the

tritium β^- decay in the blanket region. The gamma ray generated in the ^{40}K decay may be of some concern because of the long half-life of this isotope, but for ^{39}K levels of 500 ppm in lithium, the annual ^{40}K production is only around 0.03 $\mu\text{Ci}/\text{year}/\text{kg}$ of lithium.

No calculations have been performed for ^{85}Rb , ^{87}Rb , ^{79}Br , or ^{81}Br , but it should be noted that their consequences to the overall blanket neutronics could begin to become significant because of their larger neutron cross sections and more hazardous transmutation products.

Corrosion and Fabrication: The mixing and separating equipment must be constructed of materials that will not be corroded by either the lithium or the salt. Prior compatibility studies^{6,14,15} show that even the most aggressive of the fluids considered herein, i.e., liquid lithium and molten LiF , can be adequately contained in niobium base alloys. In particular, Nb-1% Zr and FS-85 (28 Ta-10.5 W-0.9 Zr-Pal. Nb) have demonstrated excellent long-term corrosion resistance in lithium^{6,14} and in LiF ¹⁵ at temperatures in excess of 900°C. Any dissolved halide species in the flowing lithium stream should be relatively benign insofar as aggravation of corrosion is concerned, because of the strongly reducing environment provided by lithium metal. This is also true for small amounts of other alkali metals dissolved in lithium.

The degree of difficulty involved in constructing the mixing and separating equipment from refractory alloys will depend to a large extent on the design of this equipment, and undoubtedly some advances in refractory-metal fabrication will be necessary. It does not appear, however, that the molten-salt extraction scheme poses fabrication requirements more stringent than those which would accrue to many of the other fluid-handling systems of currently conceived fusion reactor models.

Recovery of Tritium from the Salt: Electrochemical studies conducted at Argonne National Laboratory as part of the lithium/hydrogen galvanic cell program demonstrated that hydrogen gas, H_2 , can be electrochemically evolved from lithium hydride solutions in lithium halides. Voltametric data for $\text{Li}/\text{LiCl}/\text{H}_2$ cells^{4,16} show the onset of hydrogen evolution to be at 0.43 volt for a 5 mole % solution of LiH in LiCl-KCl and at 0.59 volt for a 0.1 mole % solution. Extrapolation of these data to an operating temperature of 500°C and a LiT concentration in the salt of $\geq 10^{-8}$ mole % indicates that, even for very dilute solutions of LiT in LiCl-KCl , the onset of tritium evolution should occur below 1.5 volts. Potentials of 1.5 volts or less will not cause any decomposition of the salt due to electrolysis, because the breakdown voltages for the salt eutectics listed in Table I (and for alkali-halide melts in general) are all greater than 2.0 volts.

The tritium gas (T_2) collection-electrode must be designed so that the evolved T_2 is recovered before appreciable back-mixing into the salt phase can occur. Several methods have been developed for the collection of electrolytically evolved gases. In particular, double

skeleton catalyst electrodes¹⁷ have been used to evolve and collect H₂ in aqueous electrolytic media. Another effective approach to gas collection at electrodes in molten salt media is the porous valve electrode described by Swinkels.¹⁸ This latter technique involves the use of an electrode assembly consisting of two porous layers, a conductive layer that is not wetted by the salt and a non-conductive layer that is wetted by the salt. Gas evolved at the interface formed by the two layers is forced through the conductive layer by the capillary pressure of the wetted insulator layer. If the nonconductive layer is on the salt-side of the electrode and the conductive layer is on the gas collection side, then the evolved gas can be collected away from the salt. Chlorine valve electrodes of this type, consisting of porous graphite (conductive layer) and a porous ceramic (nonconductive layer), have permitted reversible operation of lithium/chlorine batteries with molten LiCl electrolyte at 650°C. With proper electrode design it should be possible to develop a porous valve electrode for T₂ that can be operated in molten lithium halides.

PROCESSING CONSIDERATIONS

The solvent-extraction operation (contacting and separating) can, in principle, be carried out with various types of equipment. For the kind of liquid-liquid extraction described herein, methods based on mixer-settlers, multi-stage countercurrent-flow boxes, or centrifugal contactors would be applicable. (Reviews of the operational features and design details of these methods are given elsewhere.^{3,19,20}) All things being equal from the point of view of fabrication and operation, the centrifugal contactor approach offers the most attractive possibilities, because the short residence times involved in the centrifugal mixing and separation steps result in a minimal fluid inventory in the extraction equipment at any given time. The calculations which follow are based on a parallel network of units similar to ones described by Bernstein et al.^{3,20} Each unit is assumed to be 25-cm in diameter and 45-cm high. The height dimension includes a 10-cm weir section at the top and a 35-cm separation zone below the weir section. Each unit operates at 1700 rpm, processes 23,000 liters of fluid (lithium plus salt) per hour, and draws 3.7 kW for continuous operation.

Using the values of D_v listed in Table I, the parameters for a typical contactor unit given above, and some reasonable boundary conditions for the operating characteristics of a centrifugal contactor network, one can determine the overall processing requirements for reference model fusion reactors with liquid lithium blankets. As a case in point, we will consider the application of the centrifugal contactor method to the recently published design for the Reference Theta-Pinch Reactor²¹ (RTPR).

The main lithium circuit of the RTPR contains about 8×10^8 grams of natural lithium (7.4% lithium-6) in which approximately 60 grams per hour of tritium are bred. (The total breeding rate for the plant including a lithium-6 rich region is 87 grams of tritium per hour.) We will assume as boundary conditions that (a) the electrolytic recovery system removes a fixed fraction, ϵ , of the tritium from the salt on each pass and (b) the lithium entering each contactor unit is

mixed with and separated from an equal volume of salt. If y_i and y_o are respectively the quantities of tritium per unit time entering and leaving the bank of contactors in the lithium stream and x_i and x_o are respectively the quantities of tritium per unit time entering and leaving in the salt stream, then

$$y_i - y_o = x_o - x_i \quad (1) \quad \text{and} \quad x_i = x_o \cdot (1 - \epsilon) \quad (2)$$

As a result of the equilibrium redistribution, after contacting and separating

$$\frac{x_o}{y_o} = D_v \cdot \eta \quad (3)$$

where η is an efficiency factor ($\eta < 1$) which takes into account any mass transfer impedance to complete equilibrium. If we let R_b equal the tritium breeding rate in grams/hour, and if y_i , x_i , y_o , and x_o are also in grams/hour, then at steady-state

$$R_b = y_i - y_o = x_o - x_i = x_o \cdot \epsilon \quad (4)$$

If the tritium inventory in the blanket is uniformly distributed throughout the liquid lithium, then the fraction of the total lithium inventory that must be processed per hour, X , is simply a function of the amount of tritium that must be passed through the contactor network per hour, y_i , in order to maintain the permissible steady-state inventory of tritium in the lithium, I_{SS} , i.e.,

$$X = y_i / I_{SS} \quad (5)$$

Equations (1) through (5) can be combined to give equation (6)

$$X = \frac{R_b}{I_{SS}} \cdot \left[\frac{\epsilon \cdot D_v \cdot \eta + 1}{\epsilon \cdot D_v \cdot \eta} \right] \quad (6)$$

which permits the calculation of processing rates for fixed values of R_b , I_{SS} , D_v , η , and ϵ .

Considerations of the tritium leakage characteristics of the RTPR insofar as they affect its environmental impact have indicated that equilibrium partial pressures of tritium in the blanket region should not exceed 10^{-8} Torr, and that pressures as low as 10^{-10} Torr would be desirable. Recent thermodynamic studies of the lithium-hydrogen and lithium-deuterium systems by Veleckis et al.²² have permitted estimates to be made for the pressure-composition dependence of the lithium-tritium system in the dilute solution range. The estimated Sieverts' law behavior for dilute solutions of tritium in lithium as a function of tritium partial pressures and temperature is given by

$$C = 17.9 \cdot \sqrt{P_T} \cdot \exp(12,284/R \cdot T) \quad (7)$$

where C is the concentration of tritium in the lithium in weight ppm,

P_T is the tritium partial pressure in Torr, R is the gas constant (1.987 cal/mole $\cdot$ $^{\circ}$ K), and T is the temperature in degrees Kelvin. For $P_T = 10^{-8}$ grams, the required steady-state inventory of tritium in the lithium, I_{SS} , equals 4,250 grams, and for $P_T = 10^{-10}$ Torr, $I_{SS} = 425$ grams. (Since the lithium circuit of the RTPR operates over the temperature range 410 to 541 $^{\circ}$ C, a value of 500 $^{\circ}$ C seems to be a reasonable estimate for the average lithium temperature.)

Calculated values of the processing rates, equipment needs, and input power levels for a centrifugal contactor network designed to meet the tritium recovery requirements for the RTPR blanket system²¹ are given in Table II. These calculations show that even at the ambitiously low tritium partial pressure of 10^{-10} Torr, only about 225 contactor units (in parallel) are required to achieve steady-state tritium recovery for an effective distribution coefficient ($D \cdot \eta$) as low as 0.5 and a tritium removal efficiency in the electrolytic salt tank of 20%. In view of the overall size projected for the RTPR plant,²¹ 225 contactor units of the type described above would take up a negligible amount of space, and even for the worst case in Table II the electrical power required by the contactor network would be less than 1 MW.

The evolution of 60 grams of tritium per hour in the electrolytic salt processing tank will require 2×10^6 coulombs/hour or about 1000 amperes (at 50% current efficiency) for continuous operation. If it is assumed that a total electrode area of 10 m² is available, the resulting current density is 10 mA/cm². Since lithium/hydrogen cells have been shown⁴ to be capable of current densities in excess of 100 mA/cm², the tritium evolution process should not be current limited under these conditions. The power required to evolve the tritium at 1.5 volts is 1.5 kW--a negligible fraction of the total blanket processing power demand.

CONCLUSIONS

It is the conclusion of this study that molten salt extraction of tritium from a liquid lithium fusion reactor blanket is a feasible process. The distribution coefficients are favorable and other chemical and mechanical considerations do not indicate any serious constraints. The amounts of in-plant space and recirculating electrical power needed for continuous extraction using centrifugal contactors appear to be acceptably small. An additional benefit of such an extraction process is the potential for recovering salt-like impurity materials (e.g., oxides, carbides, nitrides, etc.) that might otherwise collect in the lithium circuits.

ACKNOWLEDGMENTS

The authors are grateful to R. R. Heinrich for assistance in the scintillation counting; to S. A. W. Gerstl for the neutronic analyses; and to M. Blander, L. Burris, and G. J. Bernstein for their comments.

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Table I. Equilibrium Distribution Coefficients for LiT Between Liquid Lithium and Selected Salt Eutectics

Eutectic Composition	Melting Point of Eutectic (°C)	Equilibration Temperature (°C)	Number of Samples	Distribution Coefficient ^a (D_v)
31 mole % LiF in LiCl	501	550 $\pm$ 50	3	1.2 $\pm$ 0.4
41 mole % KCl in LiCl	352	600 $\pm$ 50	2	2.2 $\pm$ 0.4
41 mole % RbBr in LiBr	277	500 $\pm$ 50	1	1.2

^a D_v = Tritium content per unit volume in salt/tritium content per unit volume in lithium metal

Table II. Analysis of the Molten-Salt Extraction Scheme for the RTPR Blanket System

Tritium Partial Pressure (Torr)	Effective Distribution Coefficient ($D_v \cdot \eta$)	Fraction ^a Processed Per Hour (X)	Number of Extractor Units	Required Electrical Power (MW)
10^{-10}	4.0	0.317	46	0.172
	2.0	0.494	72	0.267
	1.0	0.847	123	0.458
	0.5	1.522	225	0.839
10^{-8}	4.0	0.032	5	0.017
	2.0	0.049	7	0.027
	1.0	0.085	12	0.046
	0.5	0.155	22	0.084

^aFraction of the total lithium inventory that passes through the contactor network per hour.

Fig. 1. Schematic Diagram of a Molten-Salt Extraction Scheme for Fusion Reactors with Lithium Blankets.