The Use of Yttrium for the Recovery of Tritium
from Lithium at Low Concentrations

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ABSTRACT

A particularly difficult problem for first generation fusion power reactor systems is the recovery of tritium at very low concentration levels from the reactor blanket when lithium metal is the principal tritium breeding medium. On the basis of recent data and reasonable extrapolations, we show that it is likely that yttrium metal can be used to extract tritium from lithium at concentrations as low as 10^{-6} atom fraction tritium in lithium under conditions that are practicable for commercial power machines.

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I. INTRODUCTION

It is generally assumed that the first fusion power reactors will use deuterium and tritium as the principal fuels and that the tritium will have to be bred by capturing neutrons from the fusion reaction in a blanket containing lithium in some form.

From many standpoints the simplest and most attractive breeding medium is lithium metal. At typical temperatures proposed for fusion blankets lithium is highly fluid and thermally conductive. In addition to its high cross sections for fast and slow neutrons in tritium breeding reactions, it is inert to radiation, and the lithium density is high. However, since lithium is a good conductor of electricity, some magnetohydrodynamic energy degradations necessarily occur when the lithium is pumped across magnetic flux lines. More importantly lithium has an unusually high affinity for hydrogen and its isotopes, and consequently the recovery of tritium from lithium is difficult, especially at the low tritium concentrations required for fusion blankets.

A number of workers in the past have examined the problem of recovering tritium from lithium at low concentration levels^(1,2,3,4) and summaries of the problem have been presented by Watson⁽⁵⁾ and Johnson.⁽⁶⁾ As described below some specific recovery processes have been recommended as practicable possibilities. In this paper we review briefly the full range of recovery processes and on the basis of recent data present a critical appraisal of the likely best recovery process.

For first generation fusion power machines the principal constraint on the concentration of tritium in the reactor blanket is the total allowable tritium inventory. This inventory typically is taken to be of the order of a few kilograms,⁽⁷⁾ and since the mass of lithium in the blanket may exceed a million kilograms,⁽⁸⁾ the tolerable tritium concentration in the lithium must be around one weight part per million or less. The bulk of the tritium inventory most probably will have to be in reserve storage for the reactor fuel injection system and not in the blanket or tritium recovery system.

II. POSSIBLE SEPARATION PROCESSES

At concentration levels in the parts per million range few separation processes offer any promise of success in recovering tritium from lithium under conditions permitting easy concentration and regeneration of the tritium in pure form.

Separation processes may be classified in ascending order of cost as mechanical separations, phase-change physical separations and finally chemical processes. Mechanical processes are not applicable because the tritium in lithium at typical blanket temperatures of 600°C is present as lithium tritide dissolved in the lithium, hence is not recoverable either by filtration or by sedimentation.

Most of the phase-change separations are in principle applicable, but in practice are uneconomic. Simple cold trapping is not possible because the solubility of lithium tritide at the eutectic temperature for the lithium-lithium tritide system is

too high.⁽⁹⁾ Also at the low tritide concentrations here and temperatures near the melting point of pure lithium, the rates of crystallization would be so low that very large equipment would be required. This factor alone would also rule out the possibility of adding a solvent to the lithium to salt out the tritide, i.e., depress the solubility of the tritide below the desired concentration levels for the blanket.

Fractional distillation or indeed any vaporization process will not be practicable because an azeotrope exists for the lithium-lithium tritide system at excessively high concentrations for likely distillation temperatures. Watson⁽⁵⁾ has estimated that this azeotrope occurs at 0.05 atom ratio tritium to lithium at 1000°C. However, even in the absence of an azeotrope, or if the azeotrope were to form at sufficiently low concentrations, the vapor rates at the necessarily low pressures for distillation would lead to prohibitively large processing equipment. This factor also would preclude the use of solvent distillation (either azeotropic or extractive distillation) to break the azeotrope and effect the separation.

Liquid-liquid solvent extraction employing molten halide mixtures as solvent has been shown to be a practicable possibility by Maroni, Wolson, and Staahl.⁽¹⁰⁾ The distribution coefficient for lithium tritide between the salt melt and liquid lithium is around 2.0, and an electrolytic process for recovering the tritium from the salt has been demonstrated by Calaway.⁽¹¹⁾ Unfortunately, although the equilibrium characteristics are favorable by normal

standards, the very low concentrations dictate that the amounts of solvent be very large. Furthermore the possibilities for contamination of the lithium by the salt are unattractive.

The only important remaining phase-change separation process is adsorption, which takes advantage of a favorable distribution of the solute between the fluid phase and a concentrated phase that is physically adsorbed on the surface of an appropriate solid adsorbent. There are no obvious candidate adsorbents.

III. CHEMICAL SEPARATION PROCESSES

Only three chemical separation processes have been suggested or studied for the recovery of tritium from lithium: permeation through metal membranes; absorption by liquid hydride formers; and absorption by solid hydride formers.

We classify permeation as a chemical process because the tritium must dissociate from the tritide in order to diffuse through the metal membrane. Although permeation rates can be high and permeation processes are used industrially to extract pure hydrogen from petroleum refinery gases, the use of permeation for tritium recovery from lithium in reactor blanket systems at low tritium concentrations does not appear to be practicable. Watson⁽⁵⁾ in his early and detailed appraisal of possible means of tritium recovery from fusion blanket systems concluded that permeation might be practicable provided sufficiently thin membranes of niobium coated with palladium on the low pressure side could be used. The purpose of the palladium was to forestall oxidation of the niobium, since low tritium pressure on the down-

stream was to be achieved by maintaining a low concentration of oxygen to promote water formation. Unfortunately, it is likely that inter-diffusion of the niobium and palladium would subvert their protective features over time. Furthermore it is unlikely that nuclear construction standards would permit the use of thin enough membranes to avoid the need for very large scale equipment.

Hydride forming metals with a greater affinity for hydrogen than lithium offer another chemical means for extracting tritium from lithium. Preliminary studies on liquid-metal hydrogen getters comprising eutectic mixtures of selected rare earth metals have been undertaken by Anderson, Carstens and Alire.⁽¹²⁾ It is not clear how the tritium could be recovered from the resulting tritides, nor how contamination of the lithium by the liquid getters could be avoided or controlled easily.

As early as 1954 Salmon⁽¹³⁾ suggested the possibility of using a strong hydrogen getter like titanium or zirconium to extract tritium from lithium. However, these particular getters are at best marginal in competition with lithium. Much more attractive is yttrium, which has been studied in recent years for its application in fusion contexts by Hubberstey, Pulham and Adams⁽⁹⁾ and Clinton and Watson.⁽⁴⁾

IV. EQUILIBRIA IN LITHIUM-YTTRIUM-HYDROGEN SYSTEMS

The phase diagram for yttrium-hydrogen as adapted from Blackledge⁽¹⁴⁾ is shown in Fig. 1. At low hydrogen concentrations there is a single phase, alpha, a solid solution of hydrogen in

yttrium. With increasing hydrogen content a second phase comprising hydrogen-deficient YH_2 begins to form, and at still higher hydrogen contents hydrogen-rich YH_2 and hydrogen-deficient YH_3 begin to form. The nonstoichiometric character of the components is typical of the hydrides of transition metals. ⁽¹⁵⁾

Figure 2, based on measurements of Yannopoulos, Edwards and Wahlbeck, ⁽¹⁶⁾ shows the equilibrium hydrogen pressure exerted by various compositions of yttrium and hydrogen at various temperatures. In the temperature range 600-950°C the plateau pressures corresponding to the phase transition between yttrium and hydrogen-deficient YH_2 are given by

$$p = 4.41 \times 10^6 \exp (-27330/T) \quad (1)$$

where

p = hydrogen pressure, MPa

T = absolute temperature, kelvin.

Note in Fig. 2 that the hydrogen-deficient YH_2 phase approximates $\text{YH}_{1.8}$ over a very wide range of hydrogen pressures.

Sievert's law may be assumed to apply to the lithium-hydrogen system for low hydrogen concentrations. Recent new data of Katsuta, Ishigai and Furukawa ⁽¹⁷⁾ for the temperature range 200-450°C may be expressed

$$K_{\text{Li-H}} = \frac{C_{\text{Li}}}{p^{1/2}} = 1.196 \times 10^{-2} \exp (5342/T) \quad (2)$$

where

C_{Li} = atom fraction tritium in lithium.

At equilibrium between liquid lithium and solid yttrium the hydrogen pressures must be the same; hence for yttrium in the form of $\text{YH}_{1.8}$ the lowest equilibrium concentration of hydrogen in the

lithium will be given by the equation resulting from the elimination of p between Eqs. (1) and (2)

$$C_{Li} = 25.1 \exp (-8323/T) \quad (3)$$

Thus at 200°C the minimum achievable concentration of hydrogen in lithium using yttrium as getter is 0.57×10^{-6} atom fraction or 0.25 wppm. Of course lower concentrations could be achieved with alpha phase yttrium.

V. EQUILIBRIA IN Li-Y-T SYSTEMS

If we make the reasonable assumption that the hydrogen-tritium isotopic effect is roughly the same for the lithium-hydrogen system as it is for the yttrium-hydrogen system, the minimum achievable concentration of tritium in lithium in equilibrium contact with yttrium in the form of $YT_{1.8}$ would be the same as estimated above for the lithium-hydrogen system.

Clinton and Watson⁽⁴⁾ have measured the equilibrium characteristics of the Li-Y-T system at temperatures from 250° to 450°C at very low concentrations of tritium in yttrium, i.e., only for the alpha solid solution regime. Their results are presented as

$$K_{Y-T} = \frac{C_Y}{p^{1/2}} = 0.55e^{(22,900/RT)} \quad (4)$$

where

C_Y = wppm tritium in lithium

p = tritium pressure, torr

R = gas constant 1.987 cal/gmol, kelvin

T = absolute temperature, kelvin

Since pressures were not measured in this work, the development of Eq. (4) required information on the Sievert's law expression for the lithium-tritium system as reported by Smith et al.⁽¹⁸⁾ for 700 to 1000°C and tritium pressures 0.1 to 760 torr. This information is summed up in the equation

$$K_{\text{Li} - \text{T}} = 42.8 e^{(10,10^4/RT)} \quad (5)$$

where $K_{\text{Li} - \text{T}}$ is the Sievert's law constant, wppm/torr^{1/2}.

If we assume that the two-phase region for the tritium-yttrium system at 200°C begins at a composition equivalent to $\text{YT}_{0.25}$ and if we extrapolate Eq. (4) to that composition and also extrapolate Eq. (5) down to 200°C and pressures approaching zero, we find that the equilibrium concentration of tritium in lithium corresponding to $\text{YT}_{0.25}$ is

$$\begin{aligned} C_{\text{Li}} &= (8.41 \times 10^3 \text{ wppm}) \frac{42.8}{0.55} \exp \frac{(10104-22400)}{(1.987)(473.2)} \\ &= 0.804 \text{ wppm} \end{aligned}$$

Although this result exceeds by a factor of three the corresponding value computed for the hydrogen system, the difference is not unreasonable given the uncertainties in the data and the extrapolation into the transition region.

VI. TRANSPORT PROCESSES

Direct experimental observations of lithium-hydrogen separation using yttrium have been reported by Hubberstey, Pulham, and Adams.⁽⁹⁾ Known quantities of lithium-hydrogen solutions were kept well mixed and continuously flowing over known weights

of yttrium sponge at 400°C. The concentration of hydrogen in lithium was monitored periodically by resistivity measurements. Their data are plotted in Fig. 3 as concentration versus time.

The sudden drops in concentration at about 0.5 extraction were caused by the yttrium samples being removed from the lithium for two days during the protium experiment and for one day during the first deuterium experiment. These drops could not have occurred if surface reaction or liquid phase diffusion were rate controlling steps, since experimental conditions were not changed. The likely explanation for the breaks in the curves is the dominance of solid phase diffusion.

If diffusion in the solid is the true rate determining step, the concentration of hydrogen in the surface of the yttrium may be assumed to be in equilibrium with the bulk concentration of the hydrogen in the lithium. From Eq. (3) at 400°C the equilibrium concentration of hydrogen in lithium in contact with $\text{YH}_{1.8}$ is 0.0107 mole percent. Since this concentration was exceeded in all three of their experiments, it is reasonable to assume that the surface condition of the yttrium corresponded to $\text{YH}_{1.8}$.

In order to get a quantitative expression for the hydrogen sorption rate we write the diffusion equation for the hydrogen in the yttrium solid assuming the diffusion is unidirectional and the yttrium approximates an infinite slab.

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial z^2} \quad (6)$$

where

t = time, s

z = distance from surface in the direction of diffusion, cm

D = diffusion coefficient for hydrogen in yttrium, cm^2/s .

The initial boundary conditions are

$$\begin{array}{lll} c = 0 & 0 \leq z \leq \infty & t < 0 \\ c = 0 & z = \infty & t \geq 0 \\ c = c_0 & z = 0 & t \geq 0 \end{array} \quad (7)$$

where c_0 is the concentration of H in Y, mole/cm^3 , in equilibrium with the bulk composition of H in lithium.

For conditions (7) and constant D , Eq. (6) may be solved readily, for example by Laplace transformation, to get the familiar result ⁽¹⁹⁾

$$c = c_0 \{1 - \text{erf}(z/2\sqrt{Dt})\} \quad (8)$$

If the error function is evaluated for small values of z , Eq. (8) reduces to

$$c = c_0 (1 - z/\sqrt{\pi Dt}) \quad (9)$$

Since the rate of diffusion of the hydrogen at the surface of the solid, $\text{mol H}/\text{cm}^2\text{s}$, is

$$N_0 = -D \left(\frac{\partial c}{\partial z} \right)_{z=0} \quad (10)$$

by differentiating Eq. (9) we may express the rate as

$$N_o = c_o \sqrt{\frac{D}{\pi t}} \quad (11)$$

For a fixed volume of liquid lithium, V, and fixed surface of solid yttrium, A, the hydrogen concentration in the lithium will be governed by

$$-V \frac{\partial c_{Li}}{\partial t} = AN_o \quad (12)$$

which, with Eq. (11), may be integrated to

$$c_{Li} = c_{Li_o} - \frac{2Ac_o}{V} \sqrt{\frac{Dt}{\pi}} \quad (13)$$

where c_{Li_o} is the hydrogen concentration in the lithium at the beginning of the experiment.

On the basis of this equation we replot in Fig. 4 the original experimental data⁽²⁰⁾ as concentration of hydrogen in the lithium versus the square root of the time. The linearity of the curves is supportive of the foregoing analysis.

From Eq. (13) the slopes of the lines are $-(2Ac_o/V\sqrt{\pi})(\sqrt{D})$, and by least squares fit are equal to -0.667×10^{-3} and -1.166×10^{-3} mol percent per sec^{1/2} for hydrogen and deuterium respectively. Hubberstey et al. give for the respective yttrium surface areas estimates of 7.2 cm² and 10.1 cm² and for lithium masses 36.31 g and 25.88 g. If we take the lithium density to be 0.499 g/cm³ and the average surface composition of the yttrium to correspond to YH_{1.8}, we find with appropriate conversion of units and the values of the slopes that the respective diffusivities are

2.14×10^{-9} and $1.79 \times 10^{-9} \text{ cm}^2/\text{s}$. These values are comparable to those for hydrogen diffusivity in other rare earth metals. However, given the uncertainty in estimating effective yttrium surface areas, the accuracy probably is no better than ± 20 percent. The above analysis is based on the assumption of constant diffusivity. It can be shown that if the diffusivity is exponentially dependent on concentration, the estimated diffusivity would approximate that for the surface composition.

VII. IMPLICATIONS FOR FUSION PLANT TRITIUM RECOVERY EQUIPMENT

The plant scale tritium recovery system will comprise not fewer than three identical absorber vessels, one onstream removing tritium from the lithium, one offstream being regenerated by outgassing, and the third on standby. Heat interchangers in the lithium drag stream will reduce the lithium temperature to the 200°C operating temperature of the absorber. The design of the heat exchange system and the selection of cycle times for absorption and regeneration will require optimizations. For the purposes of this study we shall assume that a given absorber will be onstream for one day and that its regeneration can be accomplished in one day by heating the yttrium bed to sufficiently high final temperatures. Since the diffusivities of tritium in yttrium during the regeneration step will be much higher than for the sorption step, it is the latter step that will dictate the cycle times.

The high temperatures during the regeneration of the yttrium bed will also facilitate the recovery of the tritium because the permeation rates of the tritium through the absorber walls at the

likely temperatures and tritium pressures will be great enough to transfer the daily production (typically 0.5 kg tritium) into the helium purge system surrounding the absorber. Subsequent recovery of the tritium from the helium purge system can be quantitative and inexpensive. ⁽²¹⁾

VIII. ABSORBER TOWER DIAMETER

Because yttrium is costly we visualize that it will be distributed as a coating on a suitably supportive inert packing material in the form of raschig rings or berl saddles. During the absorption step the lithium drag stream will pass through this packing typically at a rate corresponding to one turnover per day of the total blanket inventory of lithium, ca. 3×10^6 kg. At a superficial velocity of 0.5 m/s with 1 cm berl saddles the absorber tower diameter would be about 0.5 m.

IX. YTTRIUM COATING THICKNESS

In order to estimate the required thickness of yttrium coating and the depth of packing we take advantage of the most convenient, available integrations of Eq. (6). Under the lithium flow conditions specified above the solid phase diffusion is the limiting rate process, and Eq. (6) provides a reasonable description for relatively thin yttrium coatings. However, the solid now is a slab of finite thickness with the boundary condition at the inside surface that the concentration gradient is always zero.

Carslaw and Jaeger⁽²²⁾ present solutions for the analogous case of heat transfer to an infinite slab of finite thickness

heated by maintaining a constant forcing temperature on both faces. The solutions are displayed as a plot of average fractional transport versus the dimensionless transport parameter $k t/l^2$, where k is the thermal diffusivity of the slab, and l is half the thickness of the slab.

For an yttrium bed initially at an average composition equivalent to $YT_{0.5}$ operated in the sorption mode to an average loading of $YT_{1.5}$ under conditions such that the exposed surface has the composition $YT_{1.8}$, the average fractional transport is $(1.5 - 0.5)/(1.8 - 0.5) = 0.77$. From the Carslaw and Jaeger figure the analogous transport parameter $Dt/l^2 = 0.51$. Thus for $D = 2 \times 10^{-9} \text{ cm}^2/\text{s}$ and 24 hours to accomplish the transport, the coating thickness l must be $(2 \times 10^{-9}(24)3600/0.51)^{1/2} = 0.018 \text{ cm}$.

X. TOWER PACKING HEIGHT

The mass of yttrium required to getter the daily tritium make of 0.5 kg for a loading from $YT_{0.5}$ to $YT_{1.5}$ is $88.9 (0.5)/3.017 = 14.7 \text{ kg}$. At a density of 4.45 g/cm^3 and an average coating thickness of 0.018 cm this mass must present a surface of $14.7 (1000)/0.018(4.45) = 1.84 \times 10^5 \text{ cm}^2$.

One-half inch berl saddles have a specific area of $142 \text{ ft}^2/\text{ft}^3(23)$ or $142/12(2.54) = 4.66 \text{ cm}^2/\text{cm}^3$. Thus the requisite packing volume is $1.84 \times 10^5/4.66 = 3.95 \times 10^4 \text{ cm}^3$ or 0.0395 m^3 , and the corresponding packing height for a tower diameter of 0.5 m is $0.0395(4)/\pi(0.5)^2 = 0.20 \text{ m}$.

XI. TRITIUM HOLDUP

Although the estimated volumes and masses for the absorber packing and yttrium are believed to be reasonable and conservative for the transport requirements, the maximum loading of the yttrium with tritium may have to be less than $YT_{1.5}$ to ensure an acceptable stability of the yttrium coating over time. If we double the estimated packing height and assume that the average tritium loading over all three absorbers (onstream, offstream and standby) approximates $YT_{0.2}$, the average tritium inventory in the absorbers will be

$$2(14.7)(3)(0.2)(3.017/88.9) = 0.6 \text{ kg} \quad .$$

XII. REMAINING PROBLEMS

It is clear that a more definitive appraisal of the utility of yttrium for tritium recovery will require accurate information (a) on thermodynamic and transport properties of yttrium - tritium-lithium systems at temperatures as low as 200°C , particularly at low tritium pressures, and (b) on the practicability of fabricating tower packing coated with yttrium metal under conditions that ensure mechanical stability on severe temperature cycling and periodic tritium loading and regeneration. The former requires new experimental data while the latter requires a development program. There appear to be no insurmountable problems at this stage, and we believe that yttrium gettering can be a practicable means for tritium recovery from lithium and that it is probably more economic than any other scheme proposed so far.

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Fig. 1. Phase Diagram of Y-H Systems (°C vs. H/Y atom ratio). 794180

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Fig. 2. H Pressure Isotherms for Y-H Systems (p (torr) vs. H/Y atom ratio).

Fig. 3. Hubberstey, Adams, Pulham Data. Mole % H in Li vs. t. 794182

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Fig. 4. Hubberstey, Adams, Pulham Data Replotted. Mole % H
in Li vs. $\sqrt{t}$.

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