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Implications of the Existing Data**

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ABSTRACT

The modeling of tritium escape from solid lithium breeders must recognize the relative importance of mass transfer in the gas phase as well as the solid-state diffusion step in the overall process. Experimental investigations on oxygen-containing compounds and intermetallics are shown to have produced considerable uncertainty in measurement of diffusion in the various materials, while data is minimal in characterizing the transfer of tritium from the gas-solid interface into the gas stream. Considerations of this latter aspect indicate important consequences for tritium inventory, tritium recovery, and the blanket design of anticipated fusion reactor systems. Future research goals that may alleviate these problems are proposed.

INTRODUCTION

The use of solid lithium compounds to breed tritium in fusion reactor blankets was initially proposed by Powell et al. (1) and Sako et al. (2) in the early 1970's and has generated considerable interest in the fusion community. The promise of these materials has led to their utilization in various forms in a number of conceptual system designs.

Under the solid breeder scheme, recovery of any bred tritium is assumed to be controlled by solid phase diffusion. Once the tritium has migrated to the solid surface, it is then swept into a purge gas stream. Recovery of the tritium easily follows upon processing of this gas. Thus, modifying conditions under which the slowest step occurs, such as decreasing the diffusion path length, can effectively decrease the amount of tritium contained in the blanket. Minimization of the tritium inventory in fusion blankets was initially the main impetus towards study of these compounds. However, the chemical stability, neutronics properties, and high melting point temperatures of these breeders have since been identified and have further promoted their development.

With recovery predicated on slow solid phase diffusion and relatively fast mass transfer to the gas phase, most experimentation in the field has concentrated on the former step. The end product of such work is either a mean time constant for release or a diffusion coefficient. It has been customary to use these experimental values in

solving the appropriate diffusion equation with zero solid-surface concentration boundary conditions to arrive at a tritium inventory estimate for the system as a whole. Overall, these efforts have found diffusion coefficients for solid breeders that suggest low inventories in the range of one to several thousand grams. The tritium inventory, under these assumptions, is formally given by

$$I = \dot{m} a^2 / 15D \quad (1)$$

where \dot{m} is the tritium mass generation rate, a is the equivalent sphere radius of the solid breeder material, and D is the tritium diffusion coefficient.

These lines of thought may be oversimplifying the problem. In order for a concentration gradient to exist at the surface and thereby provide the driving force for gas phase mass transfer, a finite surface concentration, c_s , must exist. It is then found with these considerations that the tritium inventory consists of two contributions, i.e., a term controlled by solid phase diffusion and one attributable to mass transfer in the gas phase. The inventory expression should therefore be written as

$$I = \dot{m} a^2 / 15D + M c_s \quad (2)$$

in which M represents the total mass of the breeding material. It will be shown later that in certain cases the second term may exceed the first.

An additional difficulty is recognized when conditions for the success of the sweep gas approach are examined. Little in the way of design consideration has been given to this aspect and it is often left that a "small purge gas" will suffice. Upon calculation, however, it is seen that volumetric flow rate of the purge gas may be on the same order or higher as that of the coolant. The purge gas need not be at these high pressures, of course, but the large expected pressure drops in coupling to a low pressure gas system introduce other design complications.

The intent of this paper is to review the experimental work regarding tritium release kinetics in solid breeders. A comparison of the various conditions may account for the nonconvergence of the data and suggest areas where future effort should be expended. Also, the effect of gas phase diffusion on the tritium recovery process is studied and its impact noted on the overall tritium inventory and general blanket design.

REVIEW OF THE INITIAL WORK ON TRITIUM RELEASE
KINETICS FROM SOLID LITHIUM BREEDERS

The existing data base concerning the tritium release kinetics of various solid lithium compounds has been assembled only over the last five years and is by no means conclusive. Moreover, the effort is fragmented along lines of compound type, with oxygen-containing compounds Li_2O , LiAlO_2 , Li_2SiO_3 , and Li_5AlO_4 , and intermetallics LiAl and Li_7Pb_2 each receiving significant individual study. It is found that the scope and resolution of each contributing investigation is varied, the net effect being that there are few if any common bases from which one can construct breeder comparisons.

Nonetheless, the techniques used during a typical tritium removal run to establish either a mean time constant for release, τ , or a diffusion coefficient are most often similar to those long-used to characterize fission reactor fuels. In essence, sample pellets or powders are subjected to a largely thermal flux of neutrons to generate a tritium concentration via reactions with the lithium constituents. After removal from the reactor and insertion into an extraction train, the specimen is radiometrically analyzed to yield either isochronal release curves (fraction removed vs. anneal temperature) or their isothermal counterpart (fraction removed vs. anneal time). These measurements will at least provide an estimate of τ and, if the specimen had been satisfactorily analyzed for a mean thickness or radius beforehand, will consequently lead to an estimate of D .

In addition to the specific lithium-bearing material itself, the form (particle size and morphology), pre-irradiation heat treatment, neutron influence, established tritium concentration, anneal temperature, and sweep gas constituents and flow rate all seem to be causes for the variability in experimental results. Published work in the area to date has been predominantly that of five groups. Particular experimental parameters describing these efforts are collected in Table 1.*

Fractionated powder or crushed, solidified melts of Li_3N , Li_2Si , LiAl , Li_7Pb_2 , LiAlO_2 , Li_2SiO_3 , and Li_2O have been assessed by Wiswall and Wirsing with either diffusion coefficients or mean lifetimes quoted for all but the first two materials (3,4,5). Only during runs of the aluminate and the silicate were there instances of volume diffusion clearly being the release-limiting step. Other of the tested specimens, most notably the intermetallics LiAl and Li_7Pb_2 , were conducive to mixed regime effects where bulk diffusion and solid surface-to-sweep gas mass transfer competed for overall control of the tritium escape. Particularly for these materials, a model based on bulk-diffusion is inadequate as demonstrated by the nonnegligible effects that gas flow rate, H_2 additives to the helium sweep gas, and particle size

*The tritium concentration in appm, as used in Table 1 and throughout this paper, is defined as (tritium atoms)/(total number of atoms) $\times 10^6$.

had on release behavior.

The BNL workers in addition reported favorable extraction rates for those Li_7Pb_2 and LiAlO_2 experiments conducted on pre-activation sintered samples. Apparently, phase transformation (LiAlO_2 at 900°C) and phase segregation (Li_7Pb_2) processes more than offset the consequences of a reduced specific area brought on during most powder sinterings.

The ceramics Li_2O (powder), $\beta\text{-Li}_5\text{AlO}_4$ (powders, ground spheroids), and LiAlO_2 (powders, selected fused samples) have been analyzed by Guggi and associates at Jülich (6,7,8). In most cases, grain or pellet sizes were defined to the extent that diffusion coefficients could be quoted over the applicable temperature regimes. A number of activation energies were deduced from their samples that are substantially less than those found for inert gas diffusion in UO_2 (17). However, there were no suggested mechanisms associated with these energies.

The most recent paper by the Jülich group is unique in that macroscopic mean radii were set during sample preparation. This was accomplished by grinding solidified melts into spheres of varying radii, and then using these distances to calculate diffusion coefficients from the experimentally found time constants. Because of the magnitude of these spheroids (1.67-2.88 mm diameters) relative to typical powder particle sizes, run durations were somewhat longer to achieve desired levels of tritium removal.

Investigation of the chemical species released from irradiated Li_2O powders and pellets with gas chromatographic and mass spectrometric techniques has comprised much of the work outlined in a number of papers from the Japan Atomic Energy Research Institute (JAERI) (9-13). Findings that impact directly on the potential of this oxide for fusion applications are: (1) Nearly 100% release in all samples over the anneal temperature range of $100\text{-}650^\circ\text{C}$ except in the case of high-density pellet runs where closed porosity may be leading to high retention of the isotope; (2) stabilization of recoil tritons may occur with LiOT formation and to a lesser extent HT , T_2 and HTO . Annealing at $200\text{-}600^\circ\text{C}$ brings about $\text{T}_2\text{O}(\text{g})$ or $\text{HTO}(\text{g})$ formation and release; (3) irradiation effects are suspected of being operative; runs on unirradiated LiOT materials (14) indicate an activation energy 63% larger and a pre-exponential factor some four orders-of-magnitude larger than that of the neutron-exposed specimens; (4) low percentages of HT , T_2 , CH_3T , and other hydrocarbons complete the listing of released species. The JAERI researchers performed limited size- or shape-characterizations, offering little opportunity to arrive at diffusion coefficient estimates.

Recovery of tritium from thin lithium-doped sintered aluminum product (Li-SAP) and lithium-doped aluminum (Li-Al) wafers was found to proceed under bulk diffusion control by Talbot and Wiffen at ORNL (15). Samples were fabricated from enriched ^6Li (92-96%) and were subjected to comparatively high fluences ($9.6 \times 10^{20} \text{ cm}^{-2}$ for Li-SAP

Table 1 Summary of Post-Irradiation Anneal Experiments Conducted on Leading Tritium Breeder Candidates

Material	Reference	Leading Author/ Year Reported	Sample Characterization Technique	Anneal Temperature (°C)	Gas Flow Rate Over Sample (cc/min)	Final Gas Composition	Fluence Received (cm ⁻²)	Established Tritium Conc. (appm)	Kinetics Observ. Meth.
LiAl	3,4	Wiswall/75 (BNL)	Sieving	400-600	60	9%He/91%P-10	3.6+16 ^e	1.26	PC and LSC
	15	Talbot/79 (ORNL)	Macroscopically measured	450	2000	100% Ar	9.8+19	94.8	Tritium monitor
Li ₂ Pb ₂	4	Wiswall/75	Sieving	450,550	60	9%He/91%P-10	3.6+16	1.96	PC and LSC
	5	Wiswall/77	Sieving, SEM ^b	350-550	60	9%He/91%P-10	3.6+16	1.96	PC
LiAlO ₂	3,4	Wiswall/75	Sieving	500,600,650	60	9%He/91%P-10	3.6+16	0.63	PC and LSC
	6	Guggi/75 (Jülich)	Sonic sifted	500-1150	20	50%Ar/50%CH ₄	5.7+15	0.10	PC
	6	Guggi/75	"	450-700	20	"	"	"	"
	7	Guggi/76	Manually selected	900	5	20%He/80%CH ₄	2.0+15	0.035	PC
Li ₂ O	5	Wiswall/77	Sieving & BET	650	60	9%He/91%P-10	3.6+16	0.63	PC
	16	Vasiliev/79 (USSR)	" ^f	200-800	- ^d	-	5.2+17 -2.9+18	9.1-50.	EPR
	9	Kudo/75 (JAERI)	None	100-600	30 ^d	40%He/60% ³ H ₂	3.6+16	1.68	PC, LSC, GC
	10	Tanaka/75 (JAERI)	None	100-600	30 ^d	40%He/60% ³ H ₂	3.6+16	1.68	PC, LSC, GC
Li ₂ SiO ₃	7	Guggi/76	Microscopic approx.	500-700	5	20%He/80%CH ₄	2.0+15	0.093	PC
	11,12	Masu/77 (JAERI)	Macroscop. measured	100-600	30 ^d	40%He/60% ³ H ₂	3.0+16	1.40	PC, LSC, GC
	5	Wiswall/77	Sieving, BET	500,600,650	60	9%He/91%P-10	3.6+16	1.68	PC
	13	Kudo/78	"	100-600	30	40%He/60% ³ H ₂	(2.4-3.8)+16	1.1-1.8	PC, LSC, GC
Li ₅ AlO ₄ (B-phase)	16	Vasiliev/79	"	200-450	- ^d	-	2.2+17 3.6+16	9.9-29.8	EPR
	3,4	Wiswall/75	Sieving	500	60	9%He/91%P-10	3.6+16	0.84	PC, LSC
Li ₅ AlO ₄ (B-phase)	16	Vasiliev/79	"	200-800	- ^d	-	7.8+17 2.0+18	18.-48.	EPR
	7	Guggi/76	None	400-605	5	20%He/80%CH ₄	2.0+15	0.07	PC
Li ₅ AlO ₄ (B-phase)	8	Guggi/78	Macroscopically measured	600	5	20%He/80%CH ₄	4.3+16	1.5	PC

Notes: a. The gas to the left of the slash (/) indicates the sweep gas that passes over the sample. At a later point downstream a polyatomic gas is admitted (named to the right of the slash) to improve the countability of the stream by the beta detector.
 b. Tritium concentration as given in the respective report or the maximum possible under the described experimental conditions assuming no flux distortion through the sample.
 c. Instrumentation or technique: PC - proportional counter
 LSC - liquid scintillation counter
 GC - gas chromatograph
 EPR - electron paramagnetic resonance
 BET - gaseous absorption, BET method
 SEM - scanning electron microscopy
 d. Results reported with isochronal release curves.
 e. Read as 3.6x10¹⁶.
 f. Horizontal bars (-) indicate that the particular information was unavailable.

and $9.8 \times 10^{19} \text{ cm}^{-2}$ for Li-Al). It was postulated that a nearly four orders-of-magnitude difference between derived coefficients for Li-SAP and Li-Al at 450°C may have been due to tritium trapping in the former at internal interfaces of the aluminum and Al_2O_3 particles.

Vasiliev et al. (16) have recently reported results for Li_2O , LiAlO_2 , Li_2SiO_3 , and Li_4SiO_4 powders irradiated to specific activities averaging 0.07 Ci/g ($1.4 \times 10^{18} \text{ }^3\text{T atoms/g}$). Diffusion apparently was rate-controlling for tritium release from Li_2O , but less so for the latter three compounds. Essentially complete tritium recovery was achieved in one-hour anneals at 450 , 700 , 700 , and 800°C for runs on Li_2O , Li_2SiO_3 , Li_4SiO_4 , and LiAlO_2 , respectively. Tritium fractions from Li_2O were 1% in HT or T_2 , the balance emitted as HTO and T_2O . These trends tentatively confirm the previously described JAERI findings.

Figure 1 exhibits by material and by established tritium concentration where the experimental community has concentrated its attention. Although of the eight materials, LiAlO_2 and Li_2O have attracted the most attention, the level and depth of the inquiries even for these compounds have certainly not been uniform nor complete.

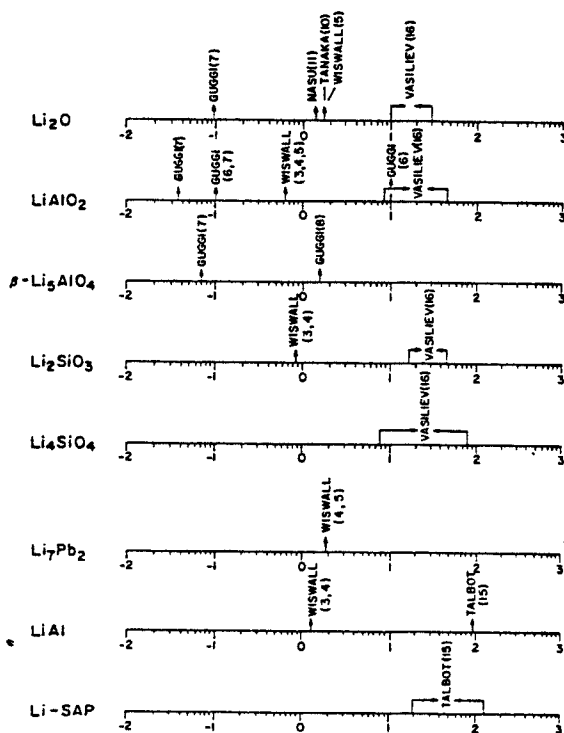


FIGURE 1 SOLID BREEDER CANDIDATE MATERIALS AND THE LOG X_1 (TRITIUM CONCENTRATION IN APPM) REGIMES IN WHICH WORKERS HAVE STUDIED SOME ASPECT OF RELEASE KINETICS. NUMBERS IN PARENTHESES INDICATE THE APPROPRIATE REFERENCE

Perhaps a more penetrating insight into the current state of knowledge regarding tritium diffusion in solid breeders may be the Arrhenius plot of diffusion coefficients, Figure 2. The data points and curves shown are taken from the work previously reviewed and cover four ceramics (LiAlO_2 , $\beta\text{-Li}_5\text{AlO}_4$, Li_2SiO_3 , and Li_2O) and three intermetallics (LiAl , Li-SAP , and Li_7Pb_2). The scatter in the laboratory derived coefficients for Li_2O , LiAlO_2 , and LiAl especially, would suggest the study of solid breeders is displaying parallels to work done on UO_2 and UC with regard to gas migration measurements, i.e., wide divergence over the probable reactor operating temperature regimes. Dissimilarities with respect to material preparation and experimental conditions among materials and groups may be the root causes.

Key aspects of this early work on fusion candidate breeder materials should be underscored:

- (1) Removal has proceeded in most materials at satisfactory rates from the small (~ 0.1 - few grams) sample sizes tested to date. Extraction curves are marked in most instances by a rapid release portion followed by a long-term, small release "tail".
- (2) The post-irradiation experiments have started with tritium concentrations near or above those expected for fusion blanket environments (~ 1 appm). However, these have been achieved with comparatively low-temperature, thermal flux irradiations. No effort has been made to separate out fast and thermal neutron damage effects.
- (3) All but a few studies have been conducted on samples that have received total integrated exposures of 10^{15} - $8 \times 10^{17} \text{ cm}^{-2}$. Recent evidence indicates that more realistic fluences (10^{18} - 10^{20} cm^{-2}) will be detrimental to the tritium release at any given anneal time (18).
- (4) Size- and shape-characterization of most samples has been limited until recently (8,15); this has resulted in large uncertainty in quoting a mean diffusion distance and consequently an even larger uncertainty in the derived diffusion coefficient.
- (5) Time constants or diffusion coefficients where they have been found are not specifically noted for a given tritium species type. The tritium may diffuse through the solid as T, T_2 , HT, HTO, T_2O , or various combinations thereof, but researchers most often do not make this distinction.

EFFECT OF GAS PHASE MASS TRANSFER ON THE TRITIUM RECOVERY PROCESS

Most calculations concerning tritium recovery from a breeding solid consider diffusion through the solid phase to be the rate-limiting step. With this premise, it is convenient in making estimates of the resulting tritium blanket inventory to assume a zero tritium concentration at the solid-gas interface. Furthermore, the additional assumption that a purge gas at adequate flow rates can be maintained in a fusion system to pick up any emitted tritium implies one need only process the gas at some later point in the flow to effect a straightforward recovery procedure. Such a tritium

in which R is the gas constant, T is the gas temperature, M is the molecular weight of the recovered species, and r denotes the mass of tritium per unit mass of tritium species recovered. Since the mass transferred out into the gas stream is proportional to $(p_s - p_g)$, a non-negative quantity, p_g should then be smaller than p_s . Therefore,

$$\dot{V} \geq \dot{m}RT/rMp_s, \quad (5)$$

and for a Li_2O breeder with a T_2O vapor pressure of $\sim 10^{-8}$ Torr at typical fusion reactor parameters of $T = 900$ K and $\dot{m} \sim 0.5$ kg/d, the minimum purge gas flow rate is 5×10^9 l/sec. If an available cross-sectional flow area of 5 m^2 exists, this would necessitate conducting the gas at an unrealistic linear velocity of 10^8 cm/sec. These considerations demonstrate that gas phase transfer cannot be neglected in assessing the likelihood of success for a tritium recovery scheme.

Experimental thermodynamic data to verify or disprove these calculations is unavailable. Yet, virtually all recovery experiments as noted earlier predict satisfactory tritium release kinetics in seeming contradiction to the above results. Figure 4 illustrates recovery rates from one set

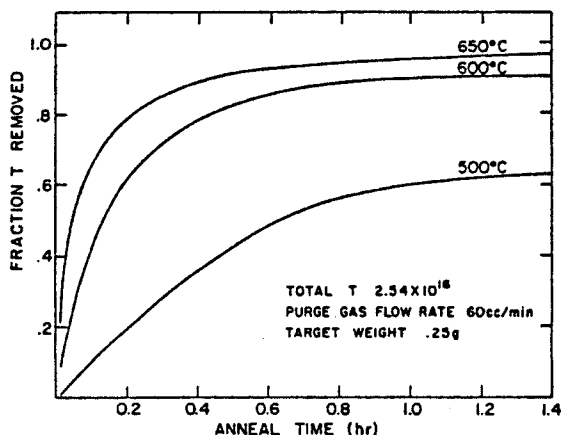


FIGURE 4 REMOVAL OF TRITIUM FROM IRRADIATED Li_2O POWDER (REPRODUCED FROM FIGURE 8. OF REFERENCE 5)

of experiments conducted on Li_2O with a sweep gas flow rate of 60 cc/min (5). Here, the oxide was dehydrated for one hour at 600–650°C. The partial pressure of T_2O in the purge gas can be calculated from the slope of the 650°C removal curve and is estimated to be 3×10^{-3} Torr at $t = 0$, or six orders-of-magnitude greater than that calculated on a theoretical basis. However, similar material to the Ventron-Alpha oxide used in the above work was analyzed by Thornton (20), who found after heat treatment a composition of 96 w/o Li_2O , 3 w/o LiOH , and 1 w/o Li_2CO_3 . Thus, it is highly probable that the tritiated form leaving the solid surface will be HTO. If the partial pressure of HTO is taken to be proportional to the H and T concentrations in the solid ($p_{\text{HTO}} \propto [\text{H}][\text{T}]$), and simi-

larly for the T_2O partial pressure at some different tritium concentration T' ($p_{\text{T}_2\text{O}} \propto [T']^2$), then an expression for $p_{\text{T}_2\text{O}}$ may be stated as

$$p_{\text{T}_2\text{O}} = p_{\text{HTO}} [T']^2 / [\text{H}][\text{T}]. \quad (6)$$

Assuming that a 3 w/o LiOH content and a 1.7 appm tritium concentration were present in the original reference, it may be seen that Equation 6 will yield $p_{\text{T}_2\text{O}} = 3 \times 10^{-6}$ Torr for dry Li_2O at one wppm tritium. This partial pressure is still much greater than the one calculated from thermodynamic data. This can be attributed to either error in the thermodynamic data or deviation from ideal solution conditions at high hydrogen concentrations. Even at this high pressure, a purge gas flow rate of larger than 2×10^7 l/sec is required to recover 0.5 kg/d of tritium. The seemingly successful recovery results reported in tritium recovery experiments may be attributed to the effect of impurities (such as H_2O) and large relative volumetric flow rates used for small samples (60 cc/min conditions are equivalent to 4×10^6 l/sec in a commercial-size reactor).

Methods have been suggested to obtain a higher recoverable tritium species' partial pressure. For example, tritium concentrations may be allowed to build up to larger values. If a design is built around a 10^5 l/sec sweep flow rate with a required tritium partial pressure of 5×10^{-4} Torr, this would demand that a Li_2O breeder contain ~ 200 appm tritium translating into an objectionably high 100 kg tritium blanket inventory. Other schemes to achieve higher partial pressures may include "wet recovery," whereby H_2O is maintained in the Li_2O so that HTO is collected instead of T_2O . Mindful of operational difficulties, the moving bed concept (21) would nevertheless allow recovery to proceed at much higher temperatures than those characteristic of the blanket. Each of the approaches has its accompanying shortcomings.

Other solid breeder compounds may offer possibly higher tritium vapor pressures that will lessen the severity of gas phase transfer effects on the overall recovery. However, experimental data is particularly lacking on these thermodynamic issues. Work with LiAl and LiAlO_2 done at BNL (4) may provide some insight. Using the 60 cc/min helium purge gas flow and 250 mg sample conditions, their release curves at 600°C indicate tritium partial pressures of 3×10^{-5} Torr and 5.6×10^{-4} Torr for LiAl and LiAlO_2 , respectively, if negligible flux distortion occurred through the samples during activation and the purge gas is saturated at early anneal times. These are reasonable working values but any optimism should be tempered as the effect of contaminants was omitted. Regardless, scaling up to a 10^5 l/sec volumetric flow rate will impact considerably on tritium recovery, tritium inventory, and blanket design planning.

CONCLUSIONS

Prediction of the overall recovery capability of tritium from any solid phase lithium compound is based on understanding the two major steps to

the escape process: (1) solid-state diffusion of the tritium species to a gas-solid interface, and (2) convective mass transfer into the purge stream. Although a number of experimental investigations on powder and pelletized samples of intermetallics and oxygen-bearing compounds have indicated generally satisfactory tritium diffusion behavior, the reported data is scattered. Tighter controls on boundary conditions (sample mean radius or thickness) and sample preparation may resolve problems with the interpretation of results. Also, these same studies have been conducted at low temperature, low thermal fluence conditions. Thus uncertainties remain when characterizing the diffusion process in materials that may likely function in a relatively high temperature, hard spectrum environment over longer exposures. Clearly, future work is warranted in these latter areas.

The second step in the recovery process is the transfer from the solid surface to the gas stream. While the kinetics of this step may be rapid, the driving force of this step, i.e., the partial pressure of the recovered tritium species may be prohibitively low. The quantity is strongly dependent on the breeding material, temperature, tritium concentration, and the presence of impurities. A low partial pressure may have a dominating effect not only on tritium inventory and recovery proposals, but on the blanket design as well. Experimental work regarding these pressure aspects is lacking. Calculations from the thermodynamic data and that obtained from tritium release experiments show a two orders-of-magnitude difference. Research on these problems must be initiated before members of the fusion community can consider one tritium handling concept as more credible over another.

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