STUDY ON THE SYSTEM EFFECT OF TRITIUM

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Tritium is sorbed not only on the surface of the specimen or material used in experiments but also on the surfaces of tubes, joints and so forth, which constitute the experimental apparatus. This could lead to incorrect experimental results and erroneous understanding of observed phenomena. The authors call this phenomenon "system effect". Therefore, it is indispensable to investigate this effect before the behavior of tritium on a certain material is studied. In this work, the system effect was studied using an experimental piping system. Moreover, numerical simulations of tritium behavior in an aluminum-coated box were carried out using results obtained in the experiments.

1. INTRODUCTION

Importance of experiments with tritium is augmenting to accumulate data required for the design of fuel cycle and safety facilities of fusion power plants as the phase of D-T fusion experiments is advancing step by step [1,2]. Tritium is adsorbed on or absorbed into most structural or piping materials of experimental equipment. For example, tritium is adsorbed on the electrode of ionization chambers, which causes the memory effect. In a previous work, we investigated the memory effect in ionization chambers and proposed a modified ionization chamber system [3]. Tritium is also sorbed on the wall of piping, joints and so forth of experimental equipment, which influences the transient response of a reactor in which an experimented sample is charged. If long tubes or components of large surface area for the sorption of tritium are used in an experimental apparatus, there is a possibility that such an experiment leads to an erroneous interpretation of phenomena. Let us call this phenomenon "system effect". The system effect also needs being examined in order to extract correct information from experimental data and also to ensure the radiation safety of fusion reactors. In this work, the authors studied the system effect using an experimental piping system. The authors also conducted a simulation study on the behavior of tritium in a glove box using data obtained in our experiment.

2. THEORETICAL BACKGROUND

If a gas containing gaseous tritium, tritiated water, protium, water and oxygen flows through a packed bed of a certain material such as breeder materials or catalysts, the following reactions take place:

(1) oxidation of gaseous tritium,
(2) adsorption of tritiated water,
(3) isotope exchange reaction between gaseous tritium in the gas stream and water on the surface of the material (let us call this reaction "exchange reaction 1"),
(4) isotope exchange reaction between tritiated water in the gas stream and water on the surface of the material (let us call this reaction "exchange reaction 2").

Thus, the mass balances in a packed bed are expressed as follows [4,5] :

・Mass balance of protium in gas stream

$$u\frac{\partial C_{H_2}}{\partial z}+\varepsilon\frac{\partial C_{H_2}}{\partial t} + K_{F,ox,H}aC_{H_2}+K_{F,ex,1}X_1=0 \quad (1)$$

・Mass balance of gaseous tritium in gas stream

$$u\frac{\partial C_{T_2}}{\partial z}+\varepsilon\frac{\partial C_{T_2}}{\partial t} + K_{F,ox,T}aC_{T_2}-K_{F,ex,1}X_1=0 \quad (2)$$

・Mass balance of water in gas stream

$$u\frac{\partial C_{H_2O}}{\partial z}+\varepsilon\frac{\partial C_{H_2O}}{\partial t} - K_{F,ox,H}aC_{H_2}+a\frac{\partial q_{net,H_2O}}{\partial t}=0 \quad (3)$$

·Mass balance of tritiated water in gas stream

$$u\frac{\partial C_{T_2O}}{\partial z}+\varepsilon\frac{\partial C_{T_2O}}{\partial t} - K_{F,ox,H}aC_{T_2}+a\frac{\partial q_{net,T_2O}}{\partial t}=0 \quad (4)$$

·Mass balance of water on surface

$$a\frac{\partial q_{H_2O}}{\partial t}-K_{F,ad}a(C_{H_2O}+C_{T_2O}-C^*)X_3 + K_{F,ex1}aX_1+K_{F,ex2}aX_2=0 \quad (5)$$

·Mass balance of tritiated water on surface

$$a\frac{\partial q_{T_2O}}{\partial t}-K_{F,ad}a(C_{H_2O}+C_{T_2O}-C^*)X_4 - K_{F,ex1}aX_1-K_{F,ex2}aX_2=0 \quad (6)$$

where

$$X_1=C_{T_2}-\frac{\left(\frac{C_{H_2}}{K}+C_{T_2}\right)q_{net,T_2O}}{q_{net,H_2O}+q_{net,T_2O}} \quad (7)$$

$$X_2=C_{T_2O}-\frac{\left(C_{H_2O}+C_{T_2O}\right)q_{net,T_2O}}{q_{net,H_2O}+q_{net,T_2O}} \quad (8)$$

when $C_{H_2O}+C_{T_2O}-C^*\geq 0$

$$X_3=\frac{C_{H_2O}}{C_{H_2O}+C_{T_2O}} \quad (9)$$

$$X_4=\frac{C_{T_2O}}{C_{H_2O}+C_{T_2O}} \quad (10)$$

when $C_{H_2O}+C_{T_2O}-C^*<0$

$$X_3=\frac{q_{net,H_2O}}{q_{net,H_2O}+q_{net,T_2O}} \quad (11)$$

$$X_4=\frac{q_{net,T_2O}}{q_{net,H_2O}+q_{net,T_2O}} \quad (12)$$

The equations shown above are related to the material balances in a packed reactor. For the system effect, related mass balance equations are shown later. Transient responses from an experimental apparatus can be calculated by combining all material balance equations.

3. EXPERIMENTAL

The experiment on the sorption of tritium was conducted by a breakthrough method with tritiated water contained in a nitrogen carrier gas. The flow sheet of the experimental apparatus is shown in Fig.1 and the experimental conditions are listed in Table 1.

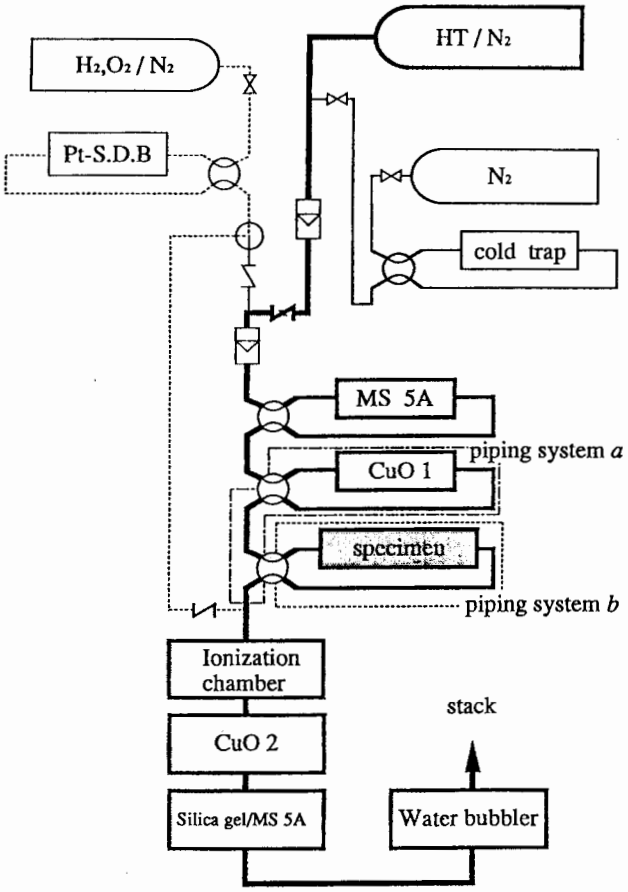

Fig.1 Flow sheet of the experimental apparatus

Table 1 Experimental conditions

Hydrogen isotopes	H_2,HT
Carrier gas	N_2
Concentration of hydrogen isotopes	20~100 ppm
Concentration of tritium	40~2200 Bq/cm³
H/T ratio $\left(\frac{H_{atom}+T_{atom}}{T_{atom}}\right)$	2000~100000
Concentration of H_2O (purge)	10000 ppm
Flow rate	0.65 *l*/min

The specimen (graphite particles or aluminum particles) was charged in a tube reactor made of quartz. The piping system of this experimental apparatus consisted of stainless steel pipes (SUS304), copper pipes and a quartz tube with brass joints. The temperature of the packed bed of specimen particles was measured with a thermocouple that was inserted in a thin quartz tube to protect it from corrosion.

The activity level of tritium in the process gas was traced using an ionization chamber with oxygen-free copper electrodes and with an effective volume of 50cm^3. The concentration of hydrogen was measured with a gas chromatograph. Water vapor was added to the process gas just before the gas was introduced into the ionization chamber in order to lower the memory effect [3].The water vapor being added to the process gas was formed in the Pt-SDB oxidation catalyst bed. A MS5A pre-adsorption bed was used to remove the residual water vapor contained in the HT/N_2 gas cylinder. A spongy CuO bed 1 was used at 300℃ to convert gaseous tritium to tritiated water.

The process gas at the exit of the ionization chamber was introduced into the spongy CuO bed 2 to oxidize gaseous tritium, and then oxidized tritium in the process gas was captured by the following silica gel/MS5A bed and water bubbler.

4. RESULTS AND DISCUSSION

4.1 EXAMPLE OF EXPERIMENTS IN WHICH SYSTEM EFFECT MUST BE CONSIDERED

As mentioned above, the system effect is induced by the sorption or the desorption of tritium or tritiated water on the piping system. Therefore, the equilibrium relationship and the mass transfer rate for sorption need to be quantified to clarify the system effects.

An example of the measurement output of the ionization chamber is shown in Fig.2, which was obtained when the sorption of tritiated water on graphite particles was investigated. In this experiment tritiated water was formed in the CuO bed (1) by oxidizing gaseous tritium at

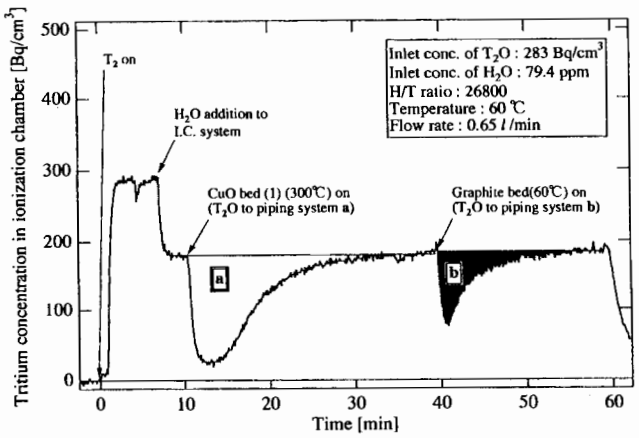

Fig.2 The example of output curve of the ionization chamber (sorption step)

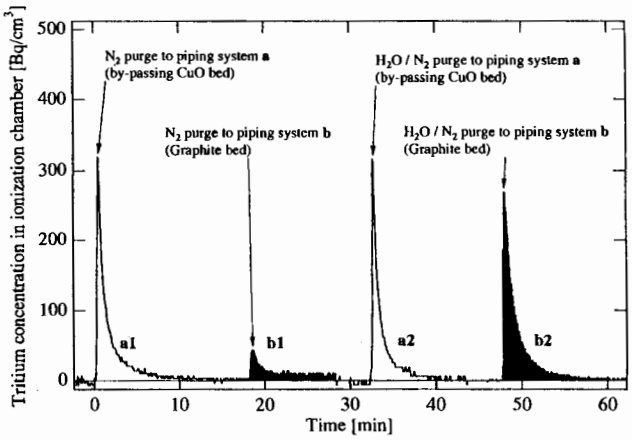

Fig.3 The example of output curve of the ionization chamber (desorption step)

Table 2 Conditions for estimation of tritium behavior in a glove box

Glove box (aluminum)	
Volume	2 m^3
Wall surface area	10 m^2
Glove box atmosphere	Ar
Sorption capacity of tritium on aluminum	2.19×10^{11} Bq/m^2
$K_{F,ex1}$	4.5×10^{-7} m/s
$K_{F,ex2}$	4.5×10^{-4} m/s
Purge gas flow rate	10 m^3/h
Temperature	20 ℃
Amount of released tritium	1.85×10^{12} Bq (100 Ci)

300C. The sorption experiments were carried out by the following procedure. First tritiated water was sorbed on the surface of the piping system **a** that consisted of the spongy CuO bed, stainless steel tube reactor, stainless steel pipes and stainless steel joints. After the sorption of tritium by the piping system **a** reached saturation, the process gas containing tritiated water was introduced to the piping system **b** which consisted of the graphite particles, quartz tube reactor, copper and stainless steal pipes and brass joints. The amount of tritium sorbed on the piping system **a** was calculated from the first breakthrough curve (a) in Fig. 2. The amount of tritium sorbed on the piping system **b** was calculated from the second breakthrough curve (b). As seen in this figure, the amount of tritium sorbed by the piping system **a** is considerably larger than that sorbed by the piping system **b** that includes the objective material (graphite) to be experimented. If this sorption experiment had been done before the sorption of tritium in the piping system **a** reached saturation, this experiment would have given a different result and resulted in a erroneous understanding of the tritium behavior over the graphite particles. It also must be noted that the amount of tritium calculated from the curve (b) in Fig.3 includes the amount of tritium sorbed on the surface of pipes and joints in the piping system **a**. Thus, this amount of tritium must be subtracted from the total amount sorbed by the piping system in order to evaluate an accurate amount of tritium sorbed on the graphite particles.

After the procedures mentioned above, tritium sorbed by piping systems **a** and **b** was purged with dry N_2 or H_2O/N_2 gases. It was first purged with the dry nitrogen gas and then purged with the H_2O/N_2 gas. The purges of sorbed tritium were separately conducted for piping systems **a** and **b**. Curves a1 and b1 in figure 3 show changes in tritium level at the exit end of each piping system when dry nitrogen gases were introduced to the piping systems: (a1) is the result of experiment for the piping system **a** and (b1) is that for the piping system **b**. After these, tritium sorbed was also purged with H_2O/N_2 gases (see curves (a2) and (b2)).

Tritium released during N_2 purges, which corresponds to (a1) and (b1) curves, is thought to be the tritium captured through adsorption on the wall of piping systems **a** and **b** during the sorption experiment (Figure 2). Tritium released during H_2O/N_2 purges (a2 and b2) could be the tritium captured through isotope exchange reactions during the sorption experiment. As shown in Figure 3, most of tritium captured on materials surface was desorbed with the water-swamped nitrogen gas whereas very small amount of tritium was desorbed with the dry nitrogen gas. These results suggest that both adsorption and isotope exchange reaction have to be considered to discuss the system effect.

As previously mentioned, it is necessary to subtract the amount of tritium sorbed on pipes or joints from the total amount of tritium captured by the piping system for the evaluation of the amount of tritium sorbed on the specimen (graphite in this experiment). In this work, the tritium sorption capacity of the experimental apparatus was quantified by a previous blank test using the experimental apparatus without specimen particles in the quartz tube.

4.2 SYSTEM EFFECT

The output curve of the ionization chamber represents not only the behavior of tritium over the specimen but also that in the piping system. In other words, the kinetic behavior of tritium over the specimen cannot be analyzed without taking into account the system effect. The over-all mass transfer coefficients for adsorption and isotope exchange reaction between tritiated water in gas phase and water on specimen were evaluated by the curve-fitting method using mass balance equations (3)~(6); equations were solved numerically.

In computations, a serial reactor model, which is schematically shown in Fig.4, was applied to the piping system **b**. In the modeling, the piping system **b** was first divided into eight serial reactors on the basis of their shapes. Next, a gas flow state was chosen for each reactor. For the gas flow state, the plug flow or the

perfectly mixed flow were considered. By this modeling, the tritium mass balance in the gas stream and that on the wall of each reactor can be expressed by the following relationship provided that the amount of adsorption of tritiated water is negligibly smaller than that of tritium captured through isotope exchange reactions.

a) plug flow reactor

·Mass balance of water in gas stream

$$u\frac{\partial C_{H_2O}}{\partial z}+\frac{\partial C_{H_2O}}{\partial t}+a\frac{\partial q_{net,H_2O}}{\partial t}=0 \qquad (13)$$

·Mass balance of tritiated water in gas stream

$$u\frac{\partial C_{T_2O}}{\partial z}+\frac{\partial C_{T_2O}}{\partial t}+a\frac{\partial q_{net,T_2O}}{\partial t}=0 \qquad (14)$$

·Mass balance of water on surface

$$a\frac{\partial q_{net,H_2O}}{\partial t}=-K_{F,ex2}aX_2 \qquad (15)$$

·Mass balance of tritiated water on surface

$$a\frac{\partial q_{net,T_2O}}{\partial t}=K_{F,ex2}aX_2 \qquad (16)$$

b) perfect mixed flow reactor

·Mass balance of water in gas stream

$$\frac{dC_{1,H_2O}}{dt}=\frac{1}{V}(\nu_0 C_{0,H_2O}-\nu_1 C_{1,H_2O})-a\frac{dq_{net,H_2O}}{dt} \qquad (17)$$

·Mass balance of tritiated water in gas stream

$$\frac{dC_{1,T_2O}}{dt}=\frac{1}{V}(\nu_0 C_{0,T_2O}-\nu_1 C_{1,T_2O})-a\frac{dq_{net,T_2O}}{dt} \qquad (18)$$

·Mass balance of water on surface

$$a\frac{dq_{net,H_2O}}{dt}=-K_{F,ex2}aX_2 \qquad (19)$$

·Mass balance of tritiated water on surface

$$a\frac{dq_{net,T_2O}}{dt}=K_{F,ex2}aX_2 \qquad (20)$$

To predict the kinetic behavior of tritium in the piping system **b**, it is necessary to obtain the sorption capacity and the over-all mass transfer coefficient in each reactor that constitute the serial reactor model. For this purpose, sorption experiments were conducted by replacing the quartz tube section in Fig. 4 with a stainless steel pipe or with a copper pipe, which were the main constituents of the piping system **b**. Most of other parts of the piping system **b** (from Cu pipe1 to Cu pipe2 in Fig.4) were also replaced with pipes made of the same material. The pipes used were 4 mm in inner diameter and 2 m in length.

Figure 5 shows an output curve of the ionization chamber obtained when a process gas was introduced into the piping system **b** of which quartz tube reactor was replaced with the stainless steel pipe. As mentioned above, this experiment was conducted after a steady state in the piping system **a** was achieved. The over-all

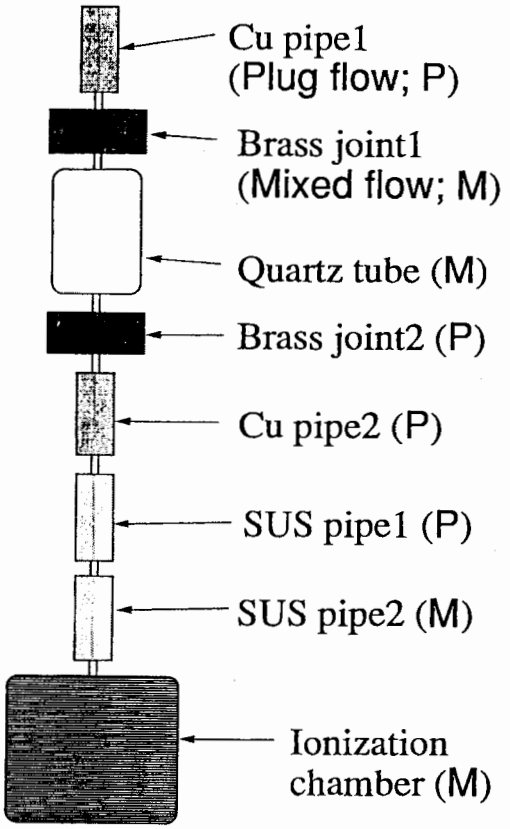

Fig.4 Serial reactor model of the piping system

mass transfer coefficient of sorption for tritium on the stainless steel was estimated to be 1.6×10^{-4} m/s. As can be seen in Fig.5, the solid line—estimated by numerical computation—agrees well with the experimental values.

The over-all mass transfer coefficient on the copper was also obtained in the same way. The over-all mass transfer coefficients obtained in this way represent the rate of isotope exchange reaction 2. This is because adsorption is negligible due to the low water vapor pressure in these experiments. In addition, the exchange reaction 2 can also be neglected, since no tritium in the molecular form is included in the process gas in these experiments.

A change in a measurement output at the ionization chamber in a case of the blank test was estimated using the over-all mass transfer coefficients and sorption property obtained in the experiments mentioned above (see Fig. 6); this computation was done with the serial reactor model. Equations (7)~(14) were used for each simulated reactor. It was assumed in the computation that the tritium sorption property for brass is the same as that for copper. It was also assumed that the quartz tube capture no tritium since its sorption capacity of tritium is much lower in comparison with stainless steel or

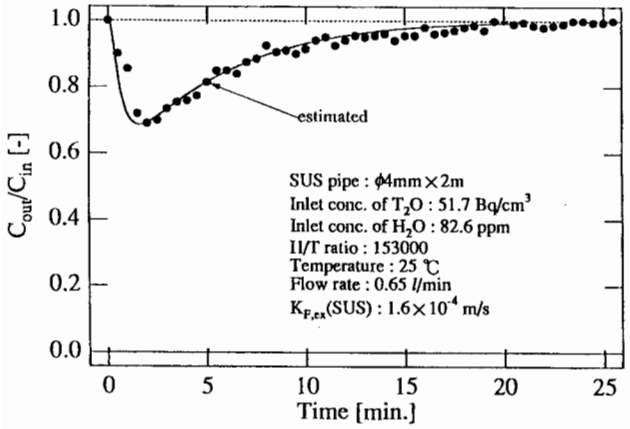

Fig.5 The output curve of the ionization chamber when piping system **b** was replaced with SUS pipe

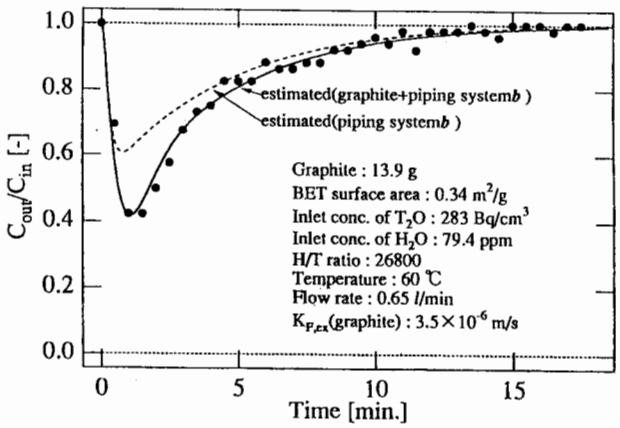

Fig.7 The output curve of the ionization chamber in piping system **b** with graphite bed

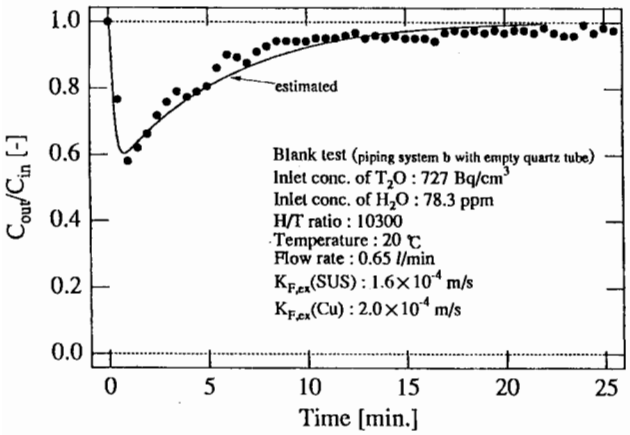

Fig.6 The output curve of the ionization chamber in piping system **b** with empty quartz tube

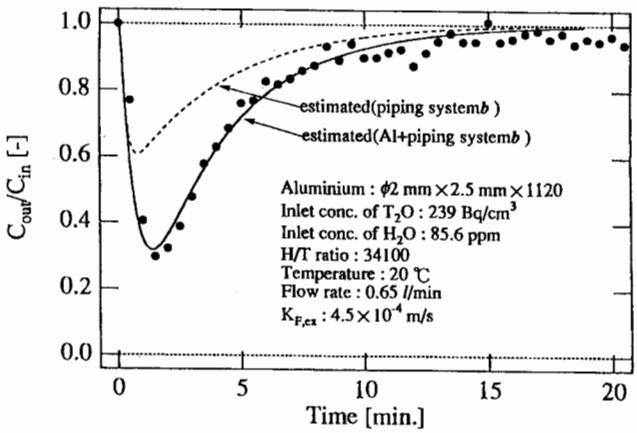

Fig.8 The output curve of the ionization chamber in piping system **b** with aluminum bed

copper [5]. As shown in Fig. 6 the estimated curve agrees well with the experimental output.

Symbols in Figure 7 show the measurement output of the ionization chamber when graphite particles were charged in the quartz tube reactor. A solid line in this figure shows the breakthrough curve calculated by the serial reactor model (the system effect is taken into account) and agrees well with the experimental result shown as closed circles. The sorption capacity of tritium on isotropic graphite, reported in a previous paper [6], was used in this computation. The broken line in this figure shows the output curve computed in the case where no graphite particles are assumed to be charged.

The output curve of the ionization chamber when aluminum particles are packed in the quartz tube (see Fig. 8) was also computed in the same way as for graphite. The sorption capacity of tritium on aluminum reported elsewhere [7] was used in this calculation.

Results shown above reveal that the behavior of tritium over some specimen can be properly predicted if the system effect is taken into consideration. It is also suggested that the serial reactor model is an effective way in computing the tritium behavior in experimental apparatus.

According to our experimental database and experiences, the following points are important to reduce the system effect in experiments:

(1) use of short pipes in the experimental apparatus
(2) use of large amounts of specimen
(3) use of materials that only capture small amounts of tritium

With regard to the system effect caused by the isotope exchange reaction, the use of tritium gas with higher H/T ratio can also decrease the system effects but the occurrence of a system effect is unavoidable since a fair amount of tritium is sorbed on almost all piping materials through adsorption, absorption or isotope exchange reactions.

In terms of the system effect, gaseous tritium has less influence because of its low adsorption capacity and slow exchange reaction rate. In our previous work, the authors evaluated the over-all mass transfer coefficient of the isotope exchange reaction 1 that takes place between gaseous tritium and surface water. This reaction is considerably slower (1/400) than the isotope exchange reaction 2 that occurs between tritiated water and the surface water [3]. However, this slow exchange reaction could inevitably lead to the system effect in the long run. It was reported that a large fraction of the tritium released from a stainless steel surface is in the form of tritiated water even when it is exposed to gaseous tritium [8,9]. Sienkiewicz also reported similar experiences for a glove box [10]. These results would support our experimental results and modeling for isotope exchange reactions.

4.3 SIMULATION STUDY

Numerical simulations of tritium behavior in a glove box were carried out by considering the system effect. A certain amount of tritium (T_2 or T_2O) was assumed to be suddenly released into a box at the initial stage. Conditions in computation are listed in Table 2. Basic assumptions in the numerical computations are

(1) The inner wall of the glove box is coated with aluminum,
(2) The gas flow in the glove box is of perfect mixed flow type,
(3) The gas purged from the glove box is not reintroduced into the box (once-through mode of operation),
(4) Only isotope exchange reactions occur (adsorption is neglected.).

The tritium sorption capacity and the over-all mass transfer coefficient for isotope exchange reaction 2 over aluminum, $K_{F,ex2}$, have been already quantified in our previous work [7]. The over-all mass transfer coefficient of isotope exchange reaction 1 on aluminum, $K_{F,ex1}$, was presumed to be 1/1000 of $K_{F,ex2}$ by consulting the previous works [3,8].

Figure 9 shows a result of numerical computations in the case where neither water nor hydrogen are contained in the purge gas. For computation, the following three cases were

considered.

Case 1: T_2O is released into the box and the sorption of tritium by the inner wall is taken into account.

Case 2: T_2 is released and the sorption of tritium by the inner wall is taken into account.

Case 3: T_2 or T_2O is released and the sorption of tritium by the inner wall is neglected.

With regard to case 3, the chemical form of tritium has nothing to do with the numerical computation because the sorption by the inner wall is neglected. The case (3) computation is an unrealistic case but we present the result of this case for the purpose of comparison with the other cases. The numbers used to distinguish the curves in this figure correspond to the numbers of the cases shown above. Curves (1), (2) and (3) show changes in the tritium concentration in the glove box (see the left vertical axis for their unit). Curves (1)' and (2)' show changes in the tritium concentration sorbed on the inner wall of the box (see the right vertical axis for their unit). It can be seen from the comparison of curves (3) and (1) that the tritium level in the glove box atmosphere decreases faster if no capacity of tritium sorption is assumed. It can be seen from curve (1)'—shown as a broken line—that the amount of tritium sorbed on the surface of the glove box wall finally becomes 1.24×10^9 Bq/m^2 (about 30 percent of the total released amount). This amount of tritium remains on the wall surface and never be removed if the purge gas does not contain H_2O or H_2. Curve (2)—representing a change in tritium level when gaseous tritium is released—almost coincides with curve (3), since the isotope exchange reaction in this case is very slow as mentioned above. In this case, tritium sorbed on the glove box wall (see the curve (2)') is only 5.97×10^9 Bq/m^2 (about 0.15 percent of released) even 120 min after the commencement of ventilation.

In the actual condition, water vapor of the order of several ppm would be present in the glove box purge gas because of a leakage through gloves. Figure 10 (a) and (b) show the effect of water vapor pressure initially contained the glove box on the transient responses of tritium levels in the glove box and on the inner wall in the case where the chemical form of

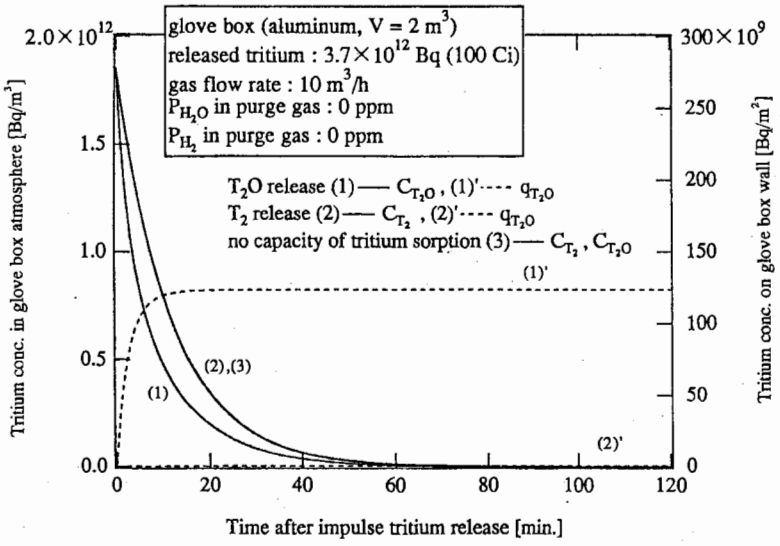

Fig.9 Tritium behavior in glove box after pulse tritium release

released tritium is tritiated water. Three cases of water vapor pressure were taken; curves (1), (2) and (3) correspond to water vapor pressure of 0, 5 and 50 ppm, respectively. The curve (4) is the result in the case where no system effect was included. As shown by curves (2) and (3) in Figs. 10 (a) and (b), tritium captured to the wall surface in the early stage—since H/T ratio in the glove box atmosphere is smaller than the ratio on the wall surface—is again released gradually to the purge gas because H/T ratio in the glove box atmosphere becomes larger than that on the surface. This result suggests that tritium once captured by the glove box wall is lately desorbed via the isotope exchange reaction 2. As the sorption capacity of tritium on the material wall is proportional to the T/H ratio as reported in the previous paper [3, 5], the addition of water vapor to the purge gas reduces the system effect. These results suggest that wet gas purge is a better choice, because it can drive out tritium from the wall surface.

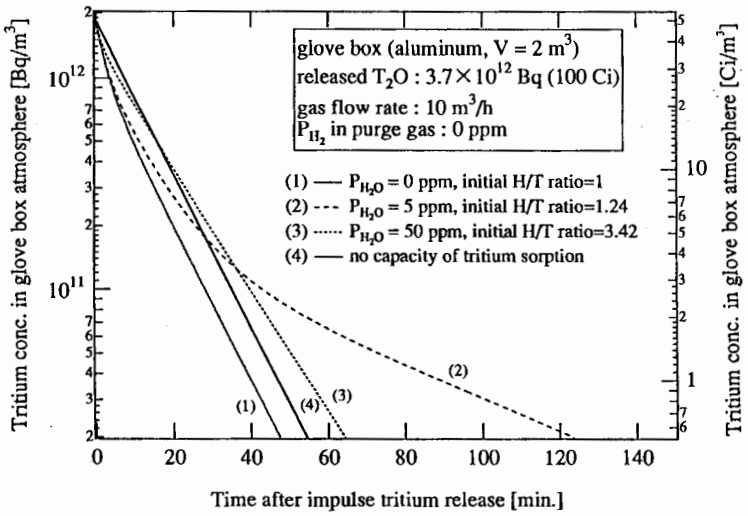

Fig.10(a) Effect of water vapor pressure on tritium behavior (concentration in gas)

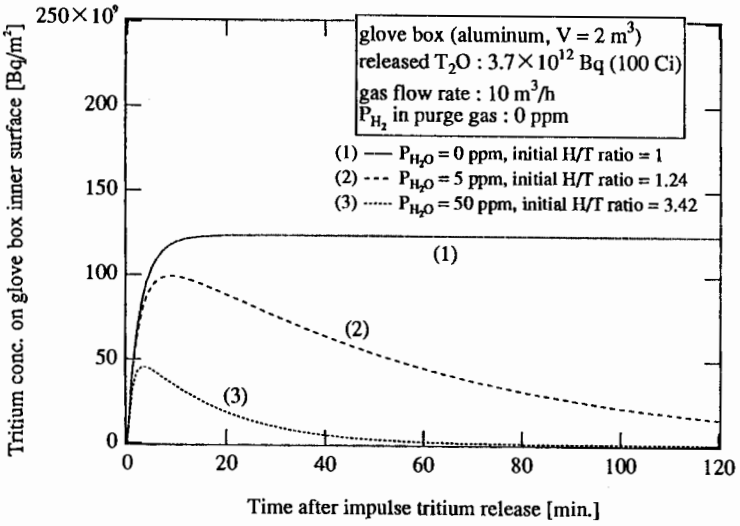

Fig.10(b) Effect of water vapor pressure on tritium behavior (concentration on wall surface)

5. CONCLUSION

The authors developed a way to evaluate the system effect based on a serial reactor model that simulates the piping system of experimental apparatus. The mass transfer rates of tritium on the experimental piping system was evaluated using the model. It was found that transient responses of a reactor which contains a reagent specimen can be properly predicted if the serial reactor model is applied and the whole mass balance equations are simultaneously solved.

Numerical simulations of tritium behavior in a glove box were also carried out using the results obtained in experiments. The results reveal that the consideration of the system effect is very important in estimating the tritium behavior in such a case.

NOMENCLATURE

a = specific surface area (m^2/m^3)
C = concentration in gas phase (mol/m^3)
C^* = equilibrium concentration of water vapor in gas phase with adsorbed water on material (mol/m^3)
ε = void fraction of the packed bed (-)
$K_{F,ox}$ = over-all mass transfer coefficient of oxidation (m/s)
$K_{F,ad}$ = over-all mass transfer coefficient of adsorption(m/s)
$K_{F,ex1}$ = over-all mass transfer coefficient of isotope exchange reaction 1 between tritium in gas phase and water on material (m/s)
$K_{F,ex2}$ = over-all mass transfer coefficient of isotope exchange reaction 2 between tritiated water in gas phase and water on material (m/s)
K = isotope effect ratio (-)
$q_{net,H2O}$ = net concentration of water on material and in sum of concentration of adsorbed water and that of structure water (mol/m^2)
$q_{net,T2O}$ = net concentration of tritiated water on material (mol/m^2)
t = time (s)
u = superficial gas velocity (m/s)
v = flow rate of process gas (m^3/s)
V = volume of reactor (m^3)
z = length in flow direction (m)
subscript
0 : inlet side
1 : inside and outlet side
init : initial

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