# Experimental Study on Signs of Ice Formation in Vacuum Drying

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## 1. Introduction

To maintain the integrity of spent nuclear fuel when storing spent nuclear fuel by dry type, canister water is drained to the outside, and the remaining water is physically bound to the canister, and spent fuel must then be dried and stored in an inert gas atmosphere.

Methods for removing residual water from dry storage system canisters include vacuum drying and forced circulation drying. The degree of drying that can be approved in the case of vacuum drying involves maintaining conditions for at least 30 min under a pressure of 3 Torr. [1].

Vacuum drying is a practical technology that has been successfully used for many years in various industries. According to a dry storage handbook and the vacuum drying test plan, ice can form during the pressure reduction phase of the vacuum drying process.

The aim of the present paper is to evaluate signs appearing that residual water inside the canister dried well as well as signs that ice had formed inside the canister during vacuum drying. To do so, a vacuum drying system and a small-sized canister were designed and fabricated. Vacuum drying tests were conducted using our vacuum drying system and canister.

### 2. Vacuum Drying Test

#### 2.1 Description of the Vacuum Drying Test Equipment

The vacuum drying test system consisted of a smallsized canister, a vacuum pump, and a cold trap (Figure 1). The body of the small-sized canister was made of stainless steel with a diameter of 406.4 mm, a length of 1,000 mm, and a thickness of 12.7 mm. The lid of the canister had a water inlet and drain port, and there was a port for installing a pressure gauge to measure pressure.



Fig. 1. Vacuum drying test equipment

### 2.2 Vacuum Drying Test

The vacuum drying test was performed seven times in total according to the following procedure:

- · Turn on the measuring equipment
- Unscrew the small-sized canister lid bolts
- Open the lid
- · Adjust the water to the desired test temperature
- Fill the beaker with 130 g water
- Place the beaker within a small-sized canister with 130 liter volume
- Close the lid
- Tighten the bolts and close the lid
- · Start the measuring equipment
- Start the test by turning on the vacuum pump
- End the test by turning off the vacuum pump

## 2.3 Test Results and Discussion

(1) Results in accordance with temperature of water

- 200 ml beaker: In the case of Test 1 (water temperature: 21.8 °C), 56 g of 130 g of water in the beaker had dried, and 74 g of water had changed into ice. In the case of Test 2 (water temperature: 12.5 °C), 53 g of 130 g of water in the beaker had dried, and 77 g changed into ice, which was about 3 g more than the ice formed in Test 1.

- 500 ml beaker: In the case of Test 3 (water temperature: 21.9 °C), 59 g of 130 g of water in the beaker had dried, and 71 g of water remained. In the case of Test 4 (water temperature: 13.6 °C), 56 g of 130 g of water in the beaker had dried, and 74 g of water remained, which was about 3 g more than in Test 3.

- 3,000 ml beaker: In the case of Test 5 (water temperature: 22 °C), 78 g of 130 g of water in the beaker had dried, and 52 g of water remained. In the case of Test 6 (water temperature: 12 °C), 75 g of 130 g of water in the beaker had dried, and 55 g of water remained, which was about 3 g more than Test 5.

During evaporation, latent heat is absorbed at a liquid surface when vapor is created. This tends to cool the surface near the interface, lowering the vapor pressure and reducing the evaporation rate [2]. Therefore, it was estimated that the lower the water temperature, the more ice formed or the more water remained.

### (2) Results in accordance with surface area of water

The inner diameter of the 200 ml, 500 ml, and 3000 ml beaker used in this study were 6 cm, 8 cm, and 15 cm, respectively. Thus, the surface area of the 200 ml, 500 ml, and 3,000 ml beakers were  $28 \text{ cm}^2$ ,  $50 \text{ cm}^2$ , and 177 cm<sup>2</sup>, respectively. After 6 hours of drying test time, if the surface area of the water was  $28 \text{ cm}^2$ , the remaining water changed to ice. However, if the surface area of the water remained.

The latent heat of vaporization of water at 0 °C is 597 cal/g [2] and the latent heat of fusion of water at 0 °C is 80 cal/g [3]. If the surface area of water is small, it is considered that ice is generated when the surrounding heat energy is exhausted because the area of the surrounding area capable of absorbing latent heat necessary for water evaporation is small. Therefore, in the vacuum drying test, the formation of ice was assessed to be related to the surface area of water.

In the test using a 200 ml beaker, 56 g (Test 1) and 53 g (Test 2) dried. In the test using a 500 ml beaker, 59 g (Test 3) and 56 g (Test 4) dried. And, in the test using a 3,000 ml beaker, 78 g (Test 5) and 75 g (Test 6) dried. Therefore, it was estimated that the wider the surface area of water, the faster the drying proceeds.





(3) Signs of ice formation and good drying

Figure 3 shows the temperature change inside the canister in accordance with the volume of the beaker.

When the residual water inside the canister dried smoothly, the internal temperature of the canister continued to decrease slightly. It was reckoned that the temperature decreases because the residual water absorbs the surrounding latent heat when it evaporates.

When ice formed inside the canister, the internal temperature of the canister continued to rise slightly. It was thought that the temperature rose because latent heat is lost to the surroundings when residual water is phase-changed into ice.

When the drying progressed smoothly inside the canister, the internal pressure of the canister was kept constant at between 4 and 5 Torr. When ice formed inside the canister, the internal pressure of the canister was maintained at slightly higher than 3 Torr.



Fig. 3. Temperature change inside the canister

Table 1. Vacuum drying test results

Test	Temp. of water (°C)	Water(After Test)			Vacuum	
		Weight(g)		Stata	Pressure	Time
		Dry	Residual	State	(Torr)	(h)
1	21.8	56	74	Ice	3.27	6
2	12.5	53	77	Ice	3.99	6
3	21.9	59	71	Water	4.24	6
4	13.6	56	74	Water	4.7	6
5	22	78	52	Water	4.62	6
6	12	75	55	Water	4.76	6
7	22	130	-	-		4.2

In Test 7, carried out by pouring 130 g of water into the canister, the internal temperature of the canister continued to decrease slightly, and the decrease stopped at about 180 min, remained a similar temperature, and the pressure and relative humidity rapidly decreased.

## 3. Conclusion

Vacuum drying tests were conducted to evaluate signs appearing that residual water inside the canister dried well as well as signs that ice had formed inside the canister during vacuum drying. The main results of our study are as follows:

- It was estimated that the lower the water temperature, the more ice had formed or the more water remained.

- The formation of ice was evaluated to be related to the surface area of water, and the wider the surface area of water, the faster the drying proceeds.

- When drying progressed smoothly inside the canister, the internal temperature of the canister continued to decrease slightly. When ice formed inside the canister, the internal temperature of the canister continued to rise slightly.

- When drying progressed smoothly inside the canister, the internal pressure of the canister was kept constant between 4 and 5 Torr. When ice formed inside the canister, the internal pressure of the canister was maintained at slightly higher than 3 Torr.

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