

# Study on Dissolution of Glass for Measurement of Activity of $\alpha$ , $\beta$ , and $\gamma$ Emitting Nuclides in Vitrification Radioactive Waste Sample

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## 1. Introduction

Nuclear power plants (NPPs) have been commercially operated in Korea since 1978, 24 NPP units being in operation and which accounting for 32% of Korean electricity generation. As a glass has advantages such as low leachability of hazardous constituents, vitrification as stabilizing of radioactive waste has been studied in the UK in the 1950s [1]. Vitrification of radioactive waste sample in Korea was tested in Hanwool NPP. As such, drums of low and intermediate level radioactive waste which include vitrification, are managed temporarily in the NPP sites. To transport radioactive waste from the temporary disposal site to the repository, > 95% of the total activity in a drum should be accounted for based on the radioactive waste acceptance criteria specified by nuclear safety and security commission (NSSC) notice No. 2015-4. Thus, we herein report the development of a dissolution procedure for radioactivity measurements of  $\alpha$ ,  $\beta$ , and  $\gamma$  ray-emitting nuclides in radioactive vitrification waste samples.

## 2. Experiments

### 2.1 Grinding of glass using a ball mill

To increase the surface area of vitrification waste sample, we made bulk sample small with a hammer and grinded the glass into small pieces using a tungsten carbide ball mill.

### 2.2 Dissolution of glass using a hot plate

The powdered inactive glass sample (0.5 g) was dissolved in the mixture of aqua regia (28 mL) with and without HF (1 mL) using a hot plate at 250°C. Following dissolution of the fresh glass sample, subsequent insoluble residue was centrifuged and filtered, respectively, using a centrifuge and a Watman No 41 filter paper. The obtained solutions were transferred to a 100 mL volumetric polypropylene (PP) flask and the volume made up to 100 mL using distilled and demineralized water (DDW). The filter paper was transferred to a Pt crucible, and then the crucible was ignited at 1000°C using an electric muffle furnace. After cooling in the desiccator, weight the crucible (labelled weight A). To measure the content of silicone dioxide by gravimetric analysis, 1:1 H<sub>2</sub>SO<sub>4</sub> (3 mL) and HF (4 mL) were added to the Pt crucible. The solution in the crucible was evaporated to dryness on a hot plate at 250°C. And then the crucible was ignited at 1000°C using an electric muffle furnace. After cooling in the desiccator, weight the crucible (labelled weight B). Subsequent insoluble residue in the crucible was dissolved in 1:1 HCl (20 mL) and HF (0.1 mL). The obtained solution was transferred to a 50 mL volumetric PP flask. Following the dissolution of inactive glass sample, the contents of constituent elements were measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES).

### 2.3 Dissolution of glass using an HP-MDS

The powdered inactive glass sample (0.5 g) was dissolved in the mixture of HCl (13.6 mL), HNO<sub>3</sub> (3.4 mL), and HF (0.6 mL) using a high-performance microwave digestion system (HP-MDS) [2]. After centrifugation, the obtained solution was transferred to a 100 mL volumetric PP flask and the volume made up to 100 mL using DDW. Subsequent insoluble residue was dissolved using an HP-MDS in the same conditions mentioned above. Following the dissolution of inactive glass sample, the contents of constituent elements were measured by ICP-AES.

### 3. Results & discussion

The powdered fresh glass sample was not dissolved in the mixture of HCl, HNO<sub>3</sub>, and HF using an HP-MDS. However, the undissolved residue was completely dissolved using the second operation of an HP-MDS in the same mixture of acids. Although the sample was completely dissolved, the analytical results of SiO<sub>2</sub> determined by ICP-AES was lower than those of certified value, owing to the volatile loss of SiF<sub>4</sub> despite the closed microwave digestion system.

On the other hand, the weight difference between A and B corresponds to the content of silicone dioxide. According to preliminary tests, the amount of dissolved Al, Ca, K, Na, and Si in the sample decreased as the amount of HF increased. Especially, 50% Na was not dissolved in the excess presence of HF. Although the loss of SiO<sub>2</sub> as SiF<sub>4</sub> happened using a hot plate, we selected a hot plate for dissolution of radioactive waste samples due to handling a lot of samples.

### 4. CONCLUSIONS

We developed a sample dissolution procedure for

the measurement of radioactivity in vitrification radioactive waste samples. The ICP-AES analytical results showed that principal constituent elements except SiO<sub>2</sub> were quantitatively recovered in dissolving sample with a mixture of aqua regia and HF on a hot plate. This procedure will be applied to vitrification radio-active waste samples for the measurement of  $\alpha$ ,  $\beta$ , and  $\gamma$  ray-emitting nuclides.

### REFERENCES

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