Preliminary Test of ^{3}H and ^{14}C Analysis Methods in Radioactive Waste

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1. Introduction

The radiochemical analysis is an essential necessity for the management and disposal of radioactive wastes. The demand of radiochemical analysis is expected to increase rapidly as the decommissioning of NPP K-1 unit which will be starting in the near future. KHNP-CRI has been building the infrastructure for the contribution of the smooth progress of decommissioning like the radiochemical analysis laboratory and analysis instruments.

In this study, separation and analytical methods for the quantitative analysis of ^{3}H and ^{14}C are investigated from radioactive waste generated from nuclear power plants. Also, preliminary tests are carried out to establish analytical methods using pyrolyser equipment, one of the analytical methods.

2. Sample pre-treatment and separation methods

2.1 Distillation method

Distillation method is one of basic methods for analyzing ^{3}H and ^{14}C. An oxidizing agent is added to the sample to be oxidized. The flask containing sample and oxidizing agent is connected to distillation unit and then the impinger. It is heated to 100-150℃ and the ^{3}H and ^{14}C as form of HTO and CO_2 are collected in the impinger. Reaction is carried out for 5 hours or more to sufficiently extract ^{3}H and ^{14}C from the sample. One of disadvantages of this method is any leakage through the connection of glasses.

2.2 Oxidation method

Oxidation method is a method to completely combust the sample at 900℃ using a combustion oven, a combustion bomb, a oxidizer, and etc. In order to select proper one of them, it is important that the separation method of the objective nuclide should be investigated through the preceding studies on the characteristics of the sample. A combustion oven is also known as tube furnace. The type and amount of sample depend on the size of the tube. The oxidation method with a combustion bomb oxidizes the sample under high pressure oxygen condition. This method takes shortly processing time. However, this method has a disadvantage that post treatment is difficult. The oxidizer method is simple and takes shortly processing time because the entire process is automated. The amount of sample is limited to 1 mg or less. It is hard to control the flowrate of gas for performing this experiment.

2.3 Separation of ^{3}H and ^{14}C in concrete samples using pyrolyser equipment

The pyrolyser equipment is composed of three furnaces, sample zone, mid zone and catalyst zone (Fig. 1). The pyrolyser should be heated to 900 ℃ for desorption of ^{3}H and ^{14}C from the sample. In the mid zone, it can be separated by condensing some volatile radionuclide (^{99}Tc, etc.) into a tube while maintaining 500℃. The catalyst zone is filled with Pt catalyst, where CO, generated by incomplete combustion, is oxidized to CO_2. The trap solution of ^{3}H and ^{14}C is 0.01 M HNO_3 and Carbo-sorb E, respectively.

Fig. 1. Configuration of the pyrolyser [1].

3. LSC calibration and sample measurement

^{3}H and ^{14}C can be measured by LSC (Liquid Scintillation Counter). Fig. 2 shows the detection efficiency curve using a CRM (Certified Reference Material). The separation is performed using simulated samples with concrete and CRM and recovery tests are measured by LSC.

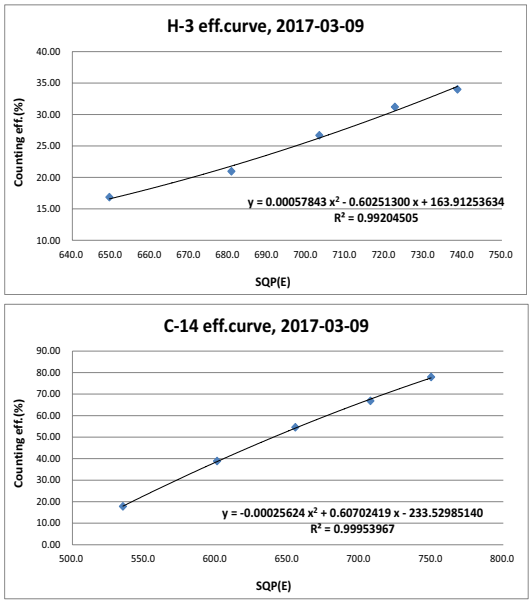

Fig. 2. Detection efficiency curve.

4. Results

The specific radioactivity, recovery rate and MDA of ^{3}H and ^{14}C are calculated from the LSC measurement results. The results are shown in table 1.

Table 1. Results of simulated sample with concrete and CRM measured by LSC

	Sample			Activity (As)	Recovery	MDA
H-3	Name	Taken(g)	Analyzed (g)	(Bq/g)	(%)	(Bq/g)
	STD 1	5.102	7.9178	3.108	91.082	0.0095
	STD 2	5.158	7.9502	3.152	92.464	0.0095

	Sample			Activity (As)	Recovery	MDA
C-14	Name	Taken(g)	Analyzed (g)	(Bq/g)	(%)	(Bq/g)
	STD 1	5.102	7.8013	1.654	96.019	0.0055
	STD 2	5.158	7.8458	1.680	98.560	0.0053

5. Conclusion

In this study, separation and analytical methods for the quantitative analysis of ^{3}H and ^{14}C are investigated from radioactive waste generated from nuclear power plants. ^{3}H and ^{14}C are analyzed by pyrolyser equipment and LSC in the simulated sample with the standard source. Good specific radioactivity, MDA and recovery rate of more than 90% are obtained.

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