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A Study on the Design of the Capture Unit of a Combustion System for Precision Analysis of Radiocarbon (^{14}C)

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1. Introduction

Radiocarbon (^{14}C) is a naturally occurring cosmic ray nuclide. It is produced by the interaction of nitrogen (^{14}N) with cosmic rays in the upper atmosphere and is widely distributed throughout the ecosystem. Because the abundance of this naturally occurring ^{14}C is very low and stable, it does not significantly impact human exposure in the general environment. However, large quantities of ^{14}C are artificially produced in nuclear power facilities during operation and fuel conversion.

The effects of ^{14}C on the human body are more important in terms of internal exposure than in general external exposure. $^{14}CO_2$ is readily absorbed into the body through respiration and, because it binds directly to biological substances in the same way as carbon metabolism in the body, can remain in the body for long periods. This process, causes tissue exposure due to the continuous emission of low-energy β-rays, posing the potential for internal exposure. Therefore, nuclear power facilities must precisely assess the amount of ^{14}C generated in exhaust gas, and accurate ^{14}C capture and analysis prior to environmental release are essential.

In particular, dry combustion-based systems can completely oxidize and collect carbon contained in the sample into CO_2, and quantifying ^{14}C using a liquid scintillation counter (LSC) is widely used. However, during the CO_2 capture process using these systems, water vapor (H_2O) in the gas can be a critical factor in reducing CO_2 capture efficiency. Moisture not only reduces the reactive activity of the CO_2 capture solution, but can also cause CO_2 to dissolve in water or destabilize the gas flow, reducing capture efficiency. The accuracy of ^{14}C analysis depends on the sample combustion system and CO_2 capture efficiency. Therefore, in this study, to improve analytical precision, designed additional capture stages within the combustion system and conducted a comparative analysis and through this, quenching characteristics and measurement efficiency were analyzed.

2. Methods and Results

High-temperature combustion is a pretreatment technique that completely oxidizes the carbon components contained in a sample at high temperatures in an oxygen (O_2) atmosphere, converting them into carbon dioxide (CO_2) gas for capture. Typically, the combustion system consists of a combustion furnace and tritium and radiocarbon capture components. The sample is combusted at high temperatures, and the generated gas passes through a catalyst layer for complete combustion. Water vapor is captured in the tritium capture element, and the remaining gas is then transferred to the ^{14}C element for capture. [1] In this study, a molecular sieve (3Å) layer was added between the tritium (^{3}H) and radiocarbon (^{14}C) capture elements to maximize the removal of moisture from the gas generated from the sample and ensure complete capture efficiency. The pore size of the 3Å molecular sieve is similar to the effective diameter of a water molecule (2.8Å), allowing it to adsorb H2O but not CO_2 (3.3Å), minimizing CO_2 loss.

2.1 Experimental Procedure

The sample to be analyzed is a charcoal filter and the sample preparation procedure is as follows:

Step 1. Weigh 0.2 g of charcoal filter granules and place them in a sample tube.

Step 2. Insert the sample tube into the combustion chamber and combust at approximately 800°C.

Step 3. During combustion, gases generated from the sample pass through the catalyst zone and are ultimately captured by the carbon absorbent.

Step 4. After combustion is complete, mix 10 ml of the solution captured in the carbon absorbent and 10 ml of the cocktail in a vial and stabilize.

Step 5. Measure the sample vial using the LSC.

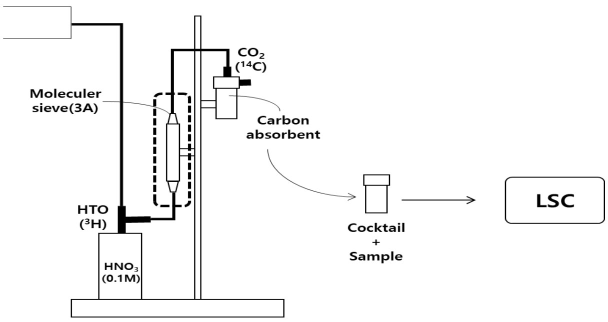

Figure 1. Sample collection diagram

2.2 Measurement and Analysis

Samples collected using the existing combustion system (^3H and ^{14}C capture stage) and a combustion system with an added moisture removal stage (3Å molecular sieve) were measured using a liquid scintillation counter (LSC). The vials used for the measurements were polyethylene vials (20 ml). To minimize measurement reproducibility and statistical error, the sample groups (#1-3) from the existing system and the sample groups (#4-6) with an added moisture removal stage system were measured ten times, each for 30 minutes. Among the measured samples, sample #6 exhibited a quenching index (tSIE) approximately 15% higher than the other samples, indicating an abnormally high measurement efficiency. This was attributed to a temporary fluctuation in sample condition during the combustion process. Therefore, it was excluded from the final statistical analysis to avoid distorting the average trends across measurement groups. Therefore, excluding sample #6, the measurement efficiency and quenching index (tSIE) for samples burned with the existing system (#1-3) and burned with the newly configured system (#4-5) were compared to examine the significance of changes in system configuration.

2.3 Experimental Results

The measurement results showed that the tSIE index and measurement efficiency of the group with the added moisture removal stage showed a slight increase compared to the existing system configuration. However, visual analysis of the standard error ranges of each data revealed that the error ranges of the two groups were close or overlapped. While the addition of the moisture removal stage was intended to improve the radiocarbon capture environment, the experimental data showed minimal differences, allowing for fluctuations within the error range. Therefore, the addition of the moisture removal stage was not sufficient to ensure statistical significance in the measurement results. To accurately assess the actual effectiveness of the moisture removal stage, quenching during measurement must be compensated for as much as possible. However, the PE vial material used in this experiment was susceptible to solvent penetration and had limitations in minimizing quenching within the sample. As a result, it appears that the physical improvement effect due to the addition of a moisture removal unit was masked by the quenching noise of the PE vial, and thus the efficiency increase due to the change in system configuration did not reach statistical significance.

Table 1. Sample measurement result (tSIE, efficiency)

Group	Sample	tSIE_avg	Unc	Eff_avg	Unc
1	#1~3	120.02	0.34	78.38	0.06
2	#4~5	123.09	0.18	78.90	0.03

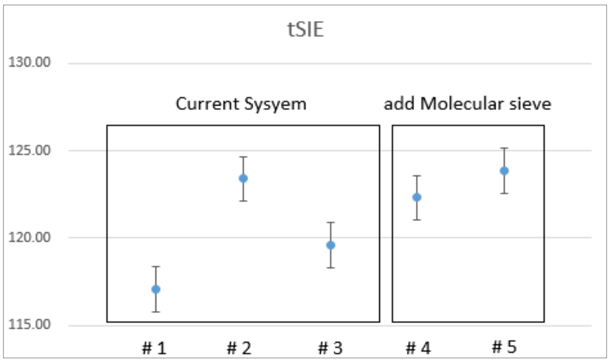

Figure 2. Quenching index(tSIE) of system composition

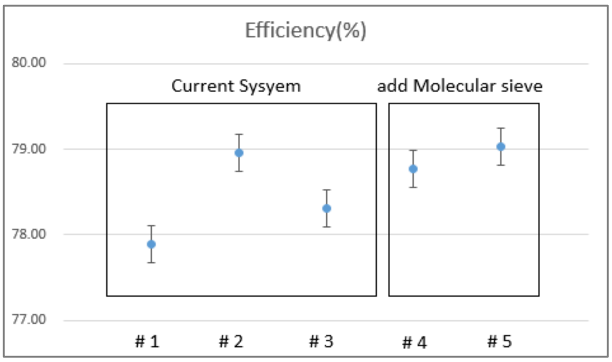

Figure 3. Measurement efficiency of system composition

3. Conclusions

This study evaluated the impact of a multi-stage capture system by adding a moisture removal stage to the existing combustion system to improve the analytical precision of ^{14}C. The experimental results confirmed an increase in measurement efficiency through the three-stage configuration. However, the standard error ranges of the data were too close to each other, making it difficult to demonstrate a statistically significant improvement. The reason for this was likely the effect of the moisture removal stage being impaired by the quenching effect during the measurement process, particularly due to the material properties of the PE vials.

To minimize the effects of quenching and more closely observe the pure effect of the moisture removal stage, future studies will re-perform the experiment using a different vial material, such as Teflon or glass. Through this, ensure the reliability of the quenching compensation and clearly verify the performance of the optimized combustion capture system.

REFERENCES

[1] Applications of Radiocarbon Isotope Ratios in Environmental Sciences in South Korea, Neung-Hwan Oh and Ji-Yeon, Seoul National University, Seoul 08826, Republic of Korea, KJEE 56(4): 281-302 (2023)