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Basic Compound Synthesis for ^{14}C Quality Verification Utilizing HANARO

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1. Introduction

^{14}C is a pure beta-emitting radioactive isotope that is the most commonly used in the form of a labeled compound with ^{3}H. In addition, ^{14}C is an isotope of carbon, a basic element of organic compounds, and be used as a radiotracer for physiological activity, metabolic tracing, and environmental change tracing in fields such as drug delivery systems, clinical research, and the environmental fields.

In particular, ^{14}C-labeled compounds are widely used in new drug development and clinical research. Microdosing is a typical example, and this technology has the advantage of being able to check human metabolism by directly injecting it into the human body.

Despite a significant amount of ^{14}C labeled compounds are being used in Korea, the production of labeled compounds is restricted because most of the basic raw materials, ^{14}C, are imported and used from abroad. To overcome this problem, it is preparing to produce ^{14}C using HANARO and will synthesize various basic compounds using ^{14}C are produced.

In this study, several experiments were conducted with cold foam to verify the quality of ^{14}C. Aluminum Nitride (AlN) not irradiated with neutrons recovers as $BaCO_3$ in carbonate form, and various basic compound synthesis experiments run using $BaCO_3$. Experimental equipment modifies to increase yield and quality, and it confirms quality through analysis equipment.

2. Methods

AlN is mainly used to produce ^{14}C through nuclear reactions in nuclear reactors. AlN has a reasonably stable structure that does not cause thermal deformation or pyrolysis following neutron irradiation.

$$^{14}N_7 + {}^{1}n_0 \rightarrow {}^{14}C_6 + {}^{1}p_1,\ \sigma=1.81b,\ t(1/2) = 5{,}370 \text{ years}$$

<$^{14}N(n,p)$ nuclear reaction>

Therefore, CO_2 generated by irradiating the AlN pellet target with neutrons was recovered in the form of $BaCO_3$, and conduct to synthesize and optimize various basic compounds, which are the previous steps of the labeling compound.

The experiment of irradiating the AlN pellet target and recovering it as $BaCO_3$ carries out as follows.

$$Al{\equiv}N + H_2SO_4 + K_2Cr_3O_7 \longrightarrow CO_2$$

<CO_2 generation process>

$$2NaOH + CO_2 \longrightarrow Na_2CO_3 + NaHCO_3$$
$$Na_2CO_3 + NaHCO_3 + Ba(OH)_2 \longrightarrow BaCO_3$$

<CO_2 capture and solidifying process>

Among the various basic compounds synthesized through $BaCO_3$, sodium acetate was selected. The synthesis was carried out as follows through the cold foam experiment.

$$BaCO_3 + H_2SO_4 \xrightarrow[Et_2O]{MeMgI} CH_3COO^{-}Na^{+}$$

A synthesis experiment was conducted based on the amount of $BaCO_3$ among the reagents used for sodium acetate synthesis. Table 1 summarizes the reagents used for the synthesis of sodium acetate. Fig 1 shows the synthesis equipment for sodium acetate.

^{1}H NMR, ^{13}C NMR is used for the analysis.

Table 1 Reagents used in the synthesis of sodium acetate

Reagents	Volume (g, ml)
$BaCO_3$ (s)	1 g
H_2SO_4 (l)	9 ml
MeMgI in ether (l)	3.2 ml
1N NaOH (l)	16.2 ml
Ag_2SO_4 (s)	1.9 g
20% H_2SO_4 (l)	8.8 ml

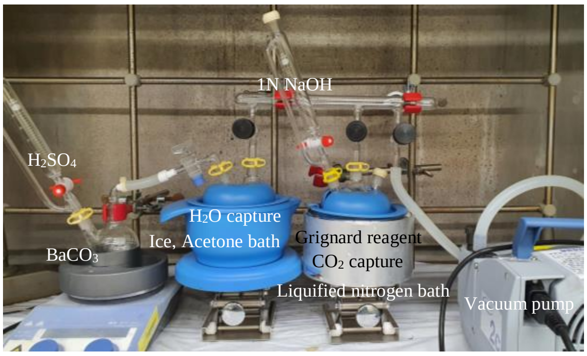

Fig 1 Sodium acetate synthesis equipment.

3. Results

The ^{14}C labeled compounds are commonly synthesized using $BaCO_3$ or basic compounds modified from $BaCO_3$. The amount of $BaCO_3$ produced from one AlN pellet target (size : ϕ 16mm, H : 8.85 mm) irradiated at HANARO was calculated 2.14 mg. And also, the amount of CO_2 gas generated from $BaCO_3$ was only 2.43 ml in STP. As such, a very small amount of ^{14}C is produced, the tightness of synthetic equipment is paramount in order to enhance the synthetic yield of the basic compound. Most of the synthetic process were carried out under vacuum, and the process optimization was carried out by changing the equipment and adjusting the number of reagents through several experiments.

At the beginning of the experiment, the yield was as low as 50%. As the experiments conduct several times, a high yield of 86% obtains by changing the experimental equipment to suppress gas leakage and increase the vacuum level. The expected yield was 0.352 g, but the amount obtained after the actual experiment was 0.342 g. The synthesis results confirm through NMR.

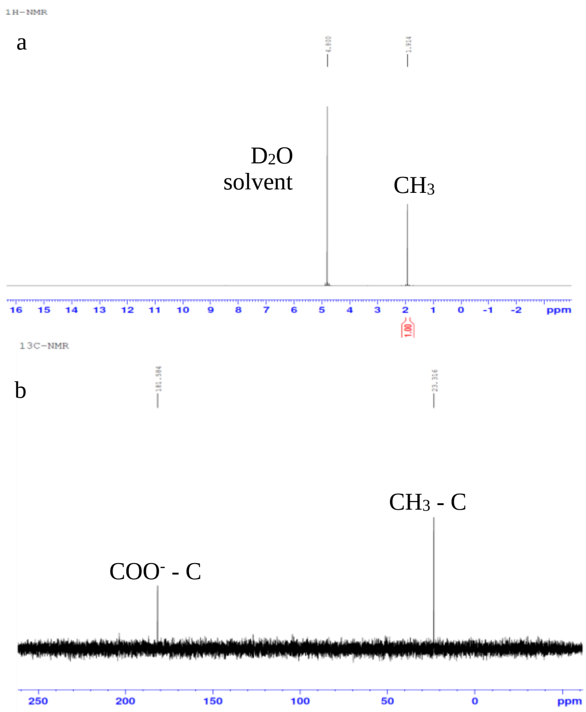

Fig 2. NMR spectrum image (a) ^{1}H NMR analysis spectrum, (b) ^{13}C NMR analysis spectrum

Through the NMR analysis confirms that the synthesis of the desirous basic compound. In addition, looking at the NMR spectrum sustains that the desired basic compound neatly synthesizes without impurities.

4. Conclusions

In this study, C was recovered in the form of $BaCO_3$, and the sodium acetate synthesis process was optimized to increase the yield and reduce waste, and the desired results were obtained. Also, the synthesis of the desired basic compound was confirmed through NMR analysis. The optimal conditions will be applied to the synthesis of basic compounds using ^{14}C which will be produced through HANARO in the future

5. Acknowledgements

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