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Key Factors in Measuring Ammonia Emissions with Dynamic Flux Chamber in Barns

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Abstract: In this study, measurement methods for estimating the NH_3 emissions in barns and the development of different emission factors were reviewed, and the factors to be considered when applying a dynamic flux chamber approach were analyzed. First, one of the factors to be considered when applying the dynamic flux chamber was determined as the stabilization time in the chamber. As a result of the experiment, it was confirmed that the concentration in the chamber stabilized after 45 min. This is considered to take longer than the stabilization time of 20 min suggested in the previous study. The second is the choice of the measurement method. This method includes real-time measurement and the indophenol method. As a result of the experiment in both methods, the ammonia flux showed a difference of about 10%, so both methods are considered to be considered. Therefore, it is judged that the methodology should be selected according to the situation, such as weather or electric power secured at the barn site. In the future, if studies on whether the stabilization time in the chamber can be changed according to seasonal factors and ambient temperature, and based on a sufficiently large sample size, the results will contribute to improving the reliability of the estimated ammonia(NH_3) emissions and the development of an emissions factor for use in the livestock sector in Korea.

Keywords: PM 2.5 secondary sources; livestock; flux chamber method; ammonia measurement method; ammonia flux

1. Introduction

1.1. Background and Purpose

The annual mean concentration of PM2.5 in Korea was 24 μg/m^3 in 2018, which ranked 27th highest among the 73 countries surveyed by AirVisual. When compared with those of Organization for Economic Cooperation and Development (OECD) member countries, the PM2.5 concentration of Korea was the second highest after Chile (24.9 μg/m^3), indicating the second poorest air quality among OECD member countries. This level of concentration was analyzed to be nearly twice as high as that of other major developed nations, such as the United States (9.0 μg/m^3), the United Kingdom (10.8 μg/m^3), Japan (12.0 μg/m^3), and France (13.2 μg/m^3) [1].

One of the causes of the increased concentration of PM2.5 is an increase in secondary products contributing to the generation of fine dust. Substances involved in the second generation of fine dust include ammonia (NH_3), nitrogen oxides (NOx), sulfur oxides (SOx), and Volatiile Organic Compounds (VOCs) [2–5]. In Korea, air pollutants have been monitored and controlled through related policies

such as the "Comprehensive Air Quality Improvement Plan" and "Regulation System on the Total Amount of Air Pollutant [6–8]." However, these policies focus on controlling the amounts of NOx and SOx among those substances contributing to the second generation of fine dust, and there have been few studies related to the identification of the sources of ammonia (NH_3) emissions or the application of emission factors when adequately considering the environment in Korea.

NH_3 emissions in Korea totaled 301,303 tons as of 2016, of which NH_3 from the agricultural sector accounted for approximately 78.7%. In the agricultural sector, the NH_3 emissions in the livestock sector amounted to 217,464 tons, accounting for approximately 92% of the NH_3 emissions in this sector, and thus an accurate estimation and management of such emissions is important [9].

Regarding the estimation of NH_3 emissions in Korea, the emission factors developed by the U.S. Environmental Protection Agency (EPA) and CO-oRdinated INformation on the Environment in the European Community-AIR (CORINAIR) in Europe have mainly been applied. In addition, there are many missing emission sources that have yet to be identified. In this regard, there is a growing need for research into the identification of such missing sources as well as the development of an NH_3 emission factor properly reflecting the characteristics of the Korean environment [10,11].

In this study, an NH_3 measurement method applicable to the estimation of NH_3 emissions in barns and the development of different emission factors are reviewed. In addition, the factors to consider when applying the widely used dynamic flux chamber method were analyzed [12].

1.2. Review of the Ammonia Measurements Methods in Barns

Barns are one of the main sources of NH_3 emissions, and large amounts of NH_3 are emitted from cattle sheds, pigsties, and poultry farms. Because the generation of NH_3 in a livestock field may vary depending on the type of barn and operation method applied, in addition to the type of livestock, the use of an appropriate measurement method is important [13,14]. Methods applicable to the measurement of NH_3 generated in barns include a tracer gas method, flux chamber method, PMU (Portable Monitoring Unit) method, and SMDAE (Saraz Method for Determination of Ammonia Emissions), descriptions of which are listed in Table 1. Among such methods, for naturally ventilated or open-type barns, tracer gas, and flux chamber methods are mainly used. In Korea, the development of an NH_3 emission factor in cattle and pig breeding facilities was calculated using a dynamic flux chamber. Therefore, in this study, the dynamic flux chamber method was also investigated, and the factors to be improved when considering the previous application of this method were analyzed.

Table 1. Description of an NH_3 measure method in a barn.

Measure Method	Description	Reference
Trace Gas method	Method of measuring flow direction and flow velocity by injecting trace gas and monitoring the concentration of trace gas overtime at the measurement location.	[15,16]
PMUs (Portable monitoring unit)	Measurement is made by continuously creating a monitoring system considering the relationship between carbon dioxide production and oxygen consumption of animals, respiratory rate (representative value), the total mass of livestock in the facility, carbon dioxide production from feces, and heat and moisture production.	[17,18]
SMDAE (Saraz Method for Determination of Ammonia Emissions)	Using a polyurethane sponge sampler (20 cm in diameter and 2 cm in thickness), a uniform mesh composed in the opposite direction was formed in the side (sidewall) opening of the building, uniformly distributed sampling, and the sampled sponge was measured for NH_3 using the Kjeldahl method.	[19,20]
Flux chamber Method	The flux chamber method is basically a method of accumulating gas discharged through the surface of a medium into space (internal chamber volume) partitioned by a researcher. There is a method to calculate the concentration change rate (dC/dt) inside the chamber over time or to calculate the concentration value after the concentration inside the chamber reaches equilibrium. In general, chambers are often used for measuring greenhouse gases (CH_4, N_2O) and Ammonia for landfills, agricultural lands or cattle farms, and mainly follow the standards of standard equipment provided by EPA.	[21–24]

2. Method

2.1. Research Design and Process

This study reviewed previous studies to identify factors for consideration when measuring NH_3 generated in a barn using a dynamic flux chamber. Pilot experiments were conducted in a laboratory after considering these identified factors and improving on them. This study also examined factors to consider in the application of actual samples, and an overview of the related system is presented in Figure 1.

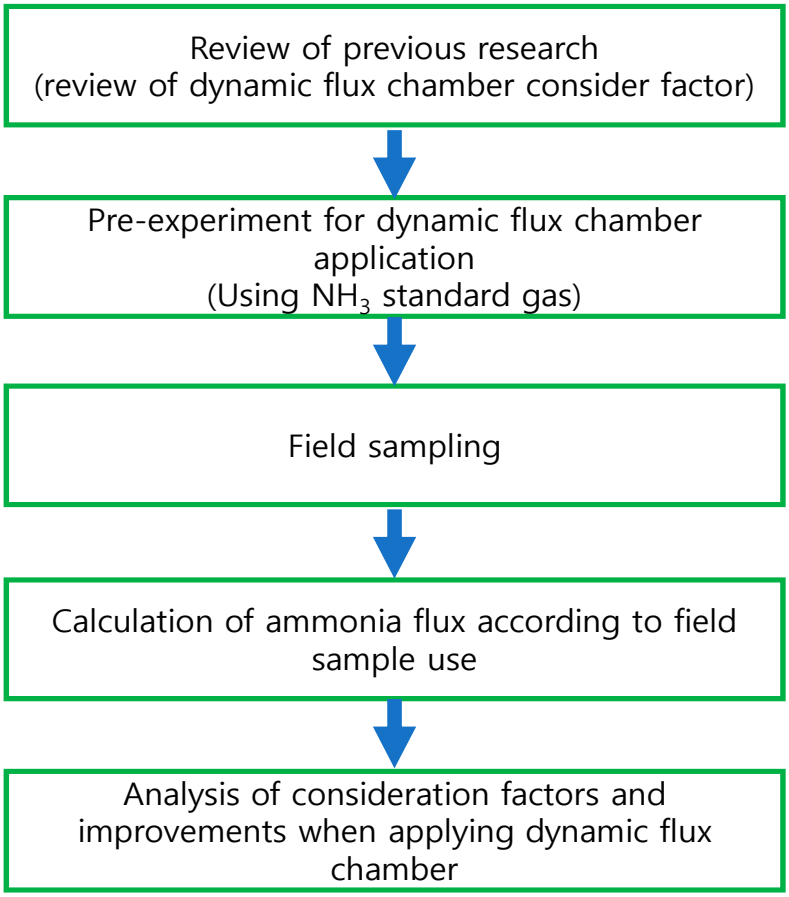

Figure 1. Process of research procedure for applying a dynamic flux chamber in a barn.

2.2. Analysis of Ammonia in a Dynamic Flux Chamber

There are two methods to measure the amount of NH_3 when using a dynamic flux chamber: The first method is a measurement after sampling the NH_3 using an indophenol-based method at the rear end of the dynamic flux chamber. The second method is a measurement of the NH_3 concentration using real-time measurement equipment at the rear end of the dynamic flux chamber. In this study, to analyze the factors to consider the application of a dynamic flux chamber in a barn, both methods for measuring NH_3 when applying a dynamic flux chamber were used for analysis.

The dynamic flux chamber was fabricated directly with reference to previous studies and the EPA method [25,26]. The size is 40.64 cm in inner diameter and 17.78 cm in height and is fabricated as an integrated type. Considering the reactivity of NH_3, the inner wall of the chamber (total volume of 30 L and a surface area of 0.13 m^2) is treated with Teflon. In the upper part, a K-type thermocouple is installed to measure the temperature inside the chamber. A pressure release (0.64 cm bulkhead union) made of Teflon is also installed to make the pressure inside the chamber equal to the atmospheric pressure, and 0.64 cm Teflon tubing is used where the gas flows in and out.

In this study, Zero Air (Ultra-high purity air 99.99999%) was used to remove various pollutants contained in the atmosphere that could affect ammonia analysis. The experimental design was made to allow Zero Air to flow into the open chamber through MFC (Mass Flow Controller) and measure the gas concentration at the outlet.

2.2.1. Ammonia Analysis Using Continuous Measuring Equipment

In this study, the amount of NH_3 was measured using a cavity ring-down spectrometer (CRDS), allowing the factors associated with the installation of continuous measurement equipment in a dynamic flux chamber to be investigated, a schematic diagram of which is shown in Figure 2. CRDS uses an analytical method that quantifies a target component by analyzing the time until the laser-irradiated inside of the analysis cell completely disappears. It is capable of a fast and continuous real-time measurement without interference and is robust against changes in ambient temperature, pressure, or vibration. However, in the case of a barn, the voltage may not be constant, and thus when using CRDS, it is necessary to check the power supply and weather effects.

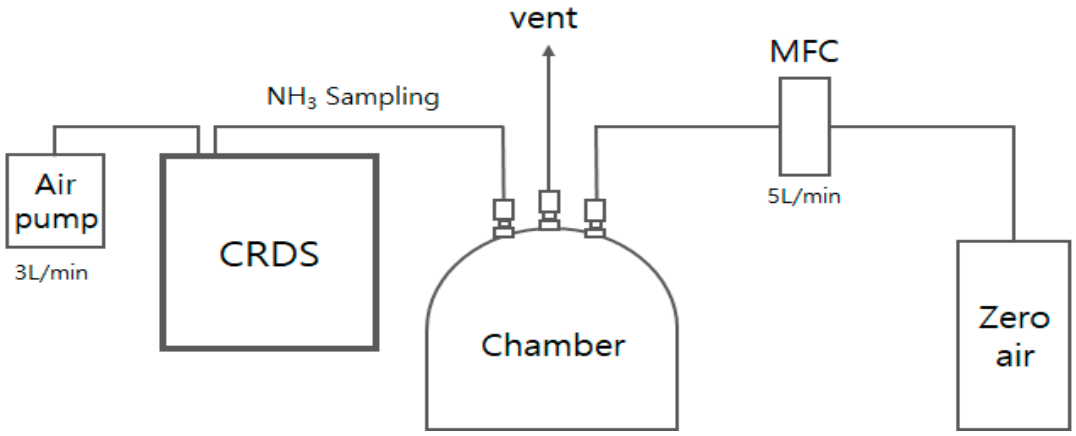

Figure 2. Schematic of the field setup using the cavity ring-down spectrometer (CRDS).

2.2.2. Ammonia Analysis Method Using Indophenol

In an NH_3 analysis method using indophenol, the amount of NH_3 is measured based on the absorbance of indophenols produced from their reaction with NH_3 ions through the addition of phenol-sodium nitroprusside and pentacyano nitrosyl iron (III) solutions to the sample solution for NH_3 analysis.

The sampling of NH_3 was conducted in accordance with Korea's "Air Pollution Process Test Method" and the "Odor Process Test Method" [27]. A schematic diagram of the experiment is shown in Figure 3. For the measurement of NH_3, and NH_3 absorbing liquid (absorption using 50 mL of a 0.5% boric acid solution) was placed in a 50 mL volumetric flask, and 45 L of gas was input at 3 L/min for approximately 15 min using a mini-pump. The inhaled sample is transferred to a volumetric flask, the total volume is 50 mL, and 10 mL of this is transferred to a test tube. To the sample solution for analysis, add 5 mL of a Sodium Pentacyanonitrosylferrate (III) Dihydrate, shake vigorously, mix 5 mL of a sodium hypochlorite solution, and stand at room temperature for 1 h. Additionally, the absorbance of the solution was measured at a wavelength of 640 nm using a spectrophotometer (Shimadzu 17A, Kyoto, Japan). The experimental method using the indophenol-based approach is shown in Figure 2. This method has the advantage of being able to adjust to the location and field situations as flexibly as possible, but it may be difficult to obtain continuous data because the process can apply only one sample every 15 min.

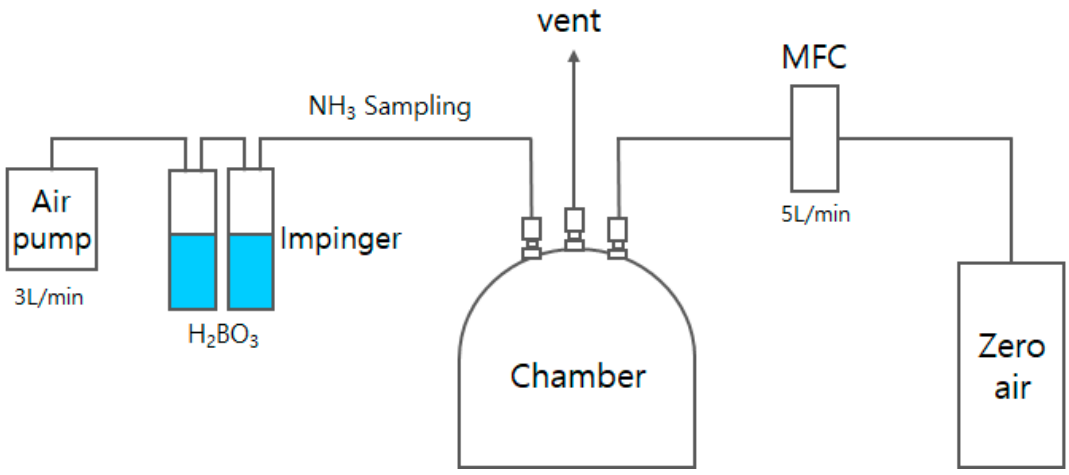

Figure 3. Schematic of the field setup using the indophenol method.

2.3. NH_3 Flux Estimation in a Barn

The method of calculating the NH_3 flux of the house was referred to as the equation presented in EPA and is as shown in Equation (1) [26].

$$Flux = \frac{V}{A_L} \times \left(L\frac{A_C}{V} + \frac{Q}{V} \right) \times C \quad (1)$$

where *Flux* is NH_3 flux in dimensions of mass per area per time (mg/m^2/min); C is NH_3 concentration in dynamic flux chamber (mg/m^3, convert ppm to mg/m^3); Q is flow rate within the dynamic flux chamber (m^3/min); A_C is surface area of the inner walls of the dynamic flux chamber above the surface (m^2); A_L is surface area covered of the dynamic flux chamber above the surface (m^2); V is volume of the dynamic flux chamber (m^3); and L is the loss term by the chamber wall per unit area assumed first order in concentration (cm/sec).

3. Results and Discussion

3.1. Estimation of Stabilization Time for Dynamic Flux Chamber Application

To determine the NH_3 concentration using a dynamic flux chamber, it is important to first determine the stabilization time and conduct the measurements using time as a parameter. Therefore, in this study, prior to the field application of a dynamic flux chamber, a preliminary experiment was conducted in the laboratory using standard NH_3 gas to determine the stabilization time for the concentration in the chamber.

The experiment was designed to apply the same conditions as those used in the field measurements, and 50 ppm (Rigas, Korea) of standard NH_3 gas was prepared through dilution with Zero Air. The target concentrations were 1 and 5 ppm. The input flow of Zero Air and the standard NH_3 gas used for dilution are shown in Table 2.

Table 2. Gas injection flow rate for stabilization time experiment.

STD (Standard Gas)	Zero Air (mL/min)	50 ppm Standard Gas (mL/min)
1 ppm	4900	100
5 ppm	4500	500

To determine the stabilization time of the NH_3 concentration in an open chamber, we conducted an experiment using CRDS, an instrument for measuring the continuous NH_3 concentration, rather than applying an indophenol-based method.

3.1.1. Stabilization of Ammonia Concentration in the Chamber According to Zero Air Input

Prior to the stabilization experiment conducted using the standard NH_3 sample as an input, another experiment was conducted on the change in NH_3 concentration in the chamber without the introduction of Zero Air, as well as on the change in the chamber after Zero Air injection.

For stabilization of the NH_3 concentration in the chamber before the Zero Air input, continuous CRDS measuring equipment was used, and a mean value of 3-min was applied after the stabilization of CRDS. It was demonstrated that the NH_3 concentration remained constant after stabilization. Stabilized ammonia concentration in the chamber before Zero Air was injected is shown in Table 3, and after stabilization, a total of 180 measurements were made for 3 min. The arithmetic mean and standard deviation were calculated using of measurement data. As the measurement results indicate, the average concentration of NH_3 in the chamber was 53 ppb, with a minimum value of 46 ppb and a maximum value of 61 ppb.

Table 3. Ammonia concentration in chamber before Zero Air injection.

Mean (ppb)	SD (Standard Deviation) (%)	Min (ppb)	Max (ppb)	*Measuring*
53	8	46	61	180

For the stabilization of the NH_3 concentration in the chamber after the input of Zero Air, a mean value of 3-min was applied after the stabilization of the CRDS, and it was observed that the NH_3 concentration reached 5 ppb, demonstrating stabilization (Figure 4). For the stabilization time of the NH_3 concentration in the chamber, 20 min after inputting Zero Air, the concentration was lowered and became stabilized. Therefore, during application at the actual site, when the NH_3 concentration falls to 5 ppb or lower with Zero Air as input, the inside of the chamber can be regarded as having been replaced with Zero Air, and it is therefore thought that this stabilization factor needs to be considered in actual field applications.

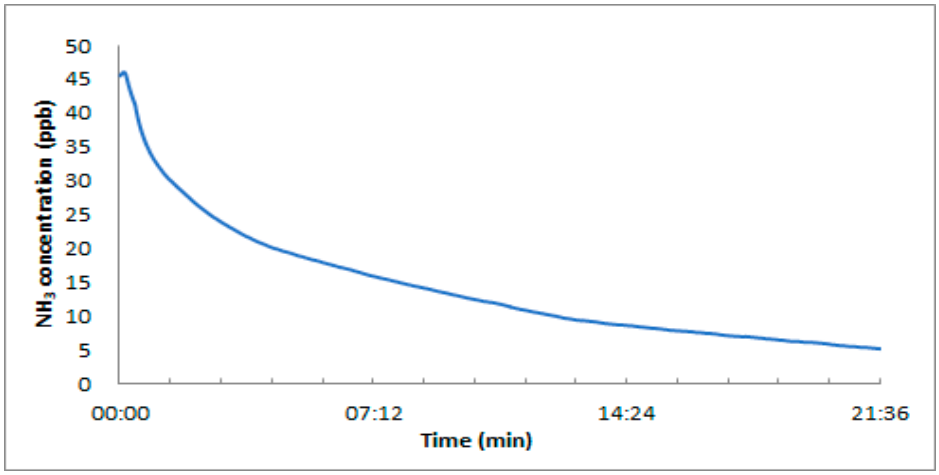

Figure 4. Stabilization time of ammonia concentration according to zero air input.

3.1.2. Stabilization of Ammonia Concentration in the Chamber According to Standard Gas

For the experiment conducted on the stabilization of the NH_3 concentration within the chamber according to the standard gas input based on a value of 5 ppm, a pre-determined flow rate (4500 mL/min for Zero Air, and 500 mL/min for NH_3 at 50 ppm) was set in each MFC, and a certain amount (2 mL/min) was first vented without passing through the chamber, and the concentration was then measured using CRDS. When the NH_3 concentration falls below 5 ppb by the Zero Air input with reference to the results of the previous experiment, the inside of the chamber is considered to have been replaced with Zero Air, after which the standard NH_3 gas is also input through the MFC. Regarding the stabilization

of the standard NH_3 gas, the moment at which the NH_3 concentration in the chamber was measured to be constant after a certain increase was thought to be the moment the inside of the chamber had become completely replaced and stabilized. The result of the measurement, shown in Figure 5, revealed that the concentration was stabilized at 4.9 ppm within approximately 45 min after injection of the 5 ppm standard NH_3 gas.

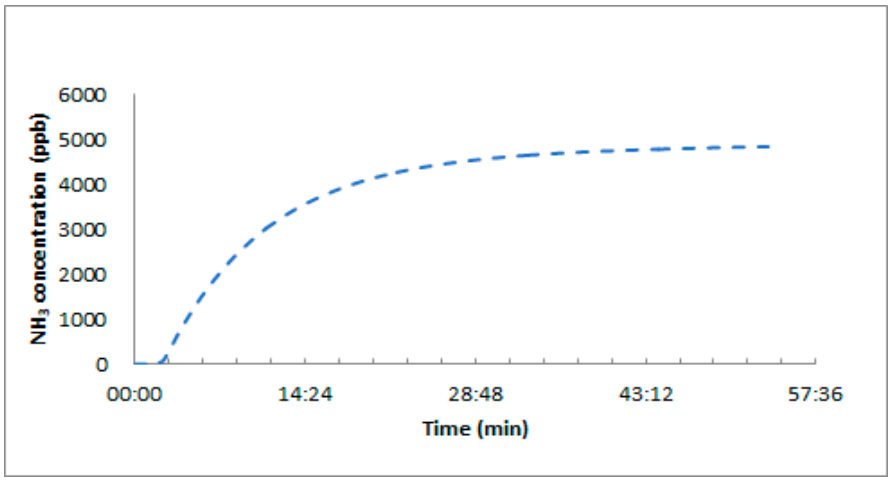

Figure 5. Stabilization time of ammonia concentration in the chamber according to STD (standard gas) 5 ppm.

The experiment conducted on the stabilization of the NH_3 concentration in the chamber according to the 1 ppm standard gas input was conducted in the same manner as that of the 5-ppm standard gas experiment after setting the pre-determined flow rate (4900 mL/min for Zero Air, and 100 mL/min for NH_3 50 ppm) in each MFC. As shown in Figure 6, the measurement results indicate that the concentration was stabilized at 1 ppm approximately 45 min after the input of 1 ppm standard NH_3 gas.

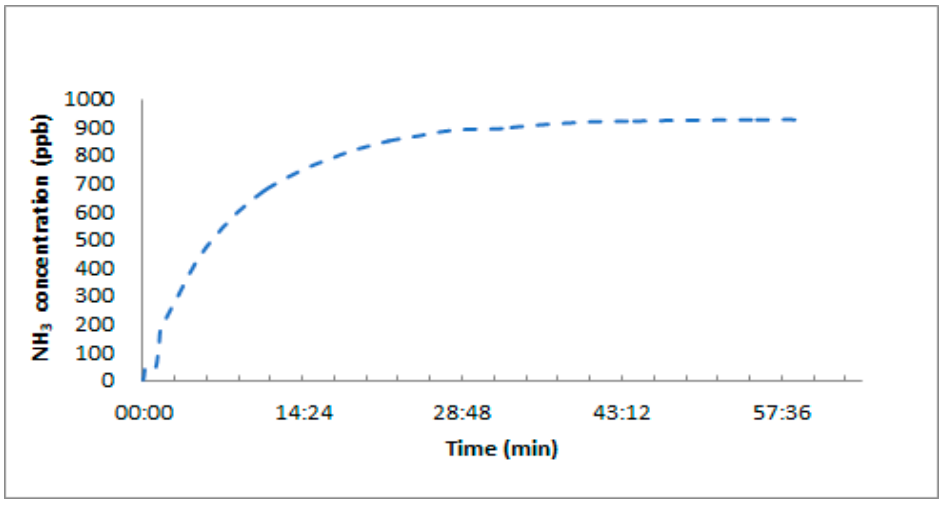

Figure 6. Stabilization time of ammonia concentration in the chamber according to STD 1 ppm.

The EPA (1986) guidelines for ammonia measurement do not mention the stabilization time in the chamber. Adviento-Borbe et al. (2010) conducted a study on ammonia and greenhouse gases in a house using a chamber, but, in the chamber, stabilization time and measurement time were not covered, and, in the case of Kim et al. (2020), the ammonia emission of liquid fertilizer was measured using a chamber, but not the stabilization time. In many of these studies, no stabilization time was mentioned. The results of the literature review, there were not many studies in other countries related

to the stabilization time of the flux chamber, so it was only possible to compare the results with the same standard in Korea. As a result of comparison with the previous studies, it can be seen that it takes longer than the stabilization time of 20 min suggested in the previous studies measured with the same standard chamber as shown in Table 4. This difference is measured by real-time measurement in this study, whereas, in the previous study, data were obtained at intervals of about 10 min using only one concentration band through the indophenol method, so it is judged as the difference between the number of data and the number of experiments. In this study, it was judged that the uncertainty may be less because experiments were conducted using two standard concentrations, the stability of multiple data and data through real-time measurement. Therefore, it is judged that the ammonia concentration can be measured only when there is a stabilization time of about 45 min for field measurement.

Table 4. Comparison of stabilization time for ammonia concentration.

This Study	NIER (2006) [28]
45 min	20 min

3.2. Calculation of NH_3 Flux in a Field Sample According to the Application of a Dynamic Flux Chamber

In this study, cattle manure samples were collected from the field, as shown in Figure 7, and experiments were conducted using a dynamic flux chamber in the laboratory.

Figure 7. Sampling of the field sample.

As shown in Figure 8, the experiment was carried out using the collected cattle manure. For the experimental procedure, Zero Air was input before the sample was added to the chamber, and once the NH_3 concentration was detected at 5 ppb or lower, the sample was added. In the case of the indophenol-based method, the NH_3 experiment was conducted after placement for more than 45 min, as in the previous experimental results. In the case of the CRDS method, it was connected directly and the stabilization time was checked.

3.2.1. NH_3 Flux of CRDS in a Dynamic Flux Chamber

The concentration of NH_3 was measured through an open chamber and CRDS in a laboratory using the manure collected from a cattle shed. The NH_3 concentration was measured by measuring the time required for the concentration to stabilize in the chamber through the CRDS after inputting the manure sample, and it was confirmed that when the field sample was used, stabilization occurred after 45 min, as shown in Figure 9, which is the same time as before. When there was no change in the NH_3 concentration in the chamber for 5 min after stabilization, the value at that time was used to

calculate the flux. After stabilization, a total of 300 measurements were made for 5 min. The arithmetic mean and standard deviation were calculated using 300 pieces of measurement data.

Figure 8. Ammonia flux estimation experiment using a field sample.

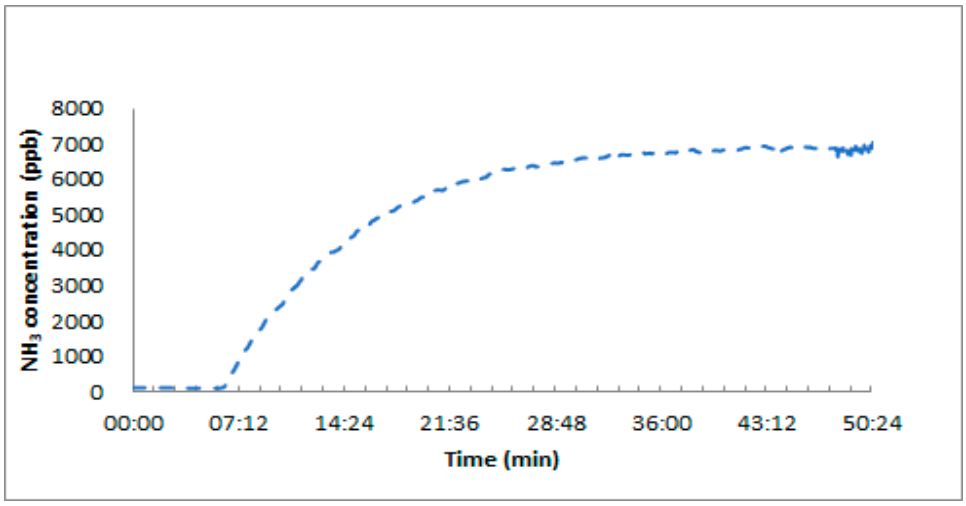

Figure 9. Ammonia concentration in an open chamber according to a field sample.

As shown in Table 5, the concentration analysis results showed a mean of 6.87 ppm, a relative standard deviation of 1%, and a concentration range of 6.65–7.05 ppm.

Table 5. Ammonia concentration of a field sample by CRDS measurement.

Mean (ppm)	SD (Standard Deviation) (%)	Min (ppm)	Max (ppm)	*Measuring*
6.87	1	6.65	7.05	300

3.2.2. NH_3 Concentration Determined Using the Indophenol-Based Method in a Dynamic Flux Chamber

The collection of NH_3 samples using the indophenol-based method was conducted after measuring the concentration stabilization time in the chamber through the CRDS after inputting the manure sample. Upon stabilization of the concentration, a total of 15 L was passed through the absorbing liquid for 5 min at 3 mL/min using a pump to calculate the NH_3 concentration value, the analysis results of which are shown in Table 6.

An analysis of the NH_3 concentration of the cattle-shed manure sample using the indophenol-based method was conducted after collecting a total of three samples. The arithmetic mean and standard

deviation were calculated using three pieces of analysis data. As a result of the analysis, the mean value was 6.25 ppm, the relative standard deviation was 0.27%, and the concentration ranged from 6.24 to 6.27 ppm.

Table 6. Ammonia concentration of a field sample by the indophenol method.

Mean (ppm)	SD (Standard Deviation) (%)	Min (ppm)	Max (ppm)	*Measuring*
6.25	0.27	6.24	6.27	3

3.2.3. Comparison of the CRDS and Indophenol Method

The CRDS and indophenol-based methods were applied in a dynamic flux chamber to analyze the target NH_3 concentration for the field sample, the results of which are shown in Table 7. As a result of the analysis, the concentration analyzed using CRDS was 6.87 ppm, and the concentration analyzed using the indophenol-based method was 6.25 ppm, which is a difference of approximately 10%. As a result of calculating the NH_3 flux according to the analysis method applied, the NH_3 flux using the CRDS method was calculated as 0.52 mg/m^2/min, and the NH_3 flux using the indophenol-based method was 0.48 mg/m^2/min, a difference in NH_3 flux of 10%, which is the same as the difference in concentration.

Table 7. Ammonia flux of a field sample according to the analysis method.

Division	CRDS	Indophenol Method
Concentration (ppm)	6.87	6.25
Flux (mg/m^2/min)	0.53	0.48

4. Conclusions

In this study, measurement methods for estimating the NH_3 emissions in barns and the development of different emission factors were reviewed, and the factors to be considered when applying a dynamic flux chamber approach were analyzed.

First, one of the factors to be considered when applying the dynamic flux chamber was determined as the stabilization time in the chamber. As a result of the experiment, it was confirmed that the concentration in the chamber stabilized after 45 min. This is considered to take longer than the stabilization time of 20 min suggested in the previous study. Therefore, it is considered that this should be taken into account when measuring.

The second is the choice of the measurement method. This method includes real-time measurement and the indophenol method. As a result of the experiment in both methods, the ammonia flux showed a difference of about 10%, so both methods can be considered. In the case of the Indophenol method, it has the advantage of being able to cope with the location and field situation as flexibly as possible, but it is difficult to obtain continuous data by taking one sample per 15 min, and the degree of stabilization of the concentration in the chamber cannot be checked in real-time. Real-time measurement equipment has an advantage in that data can be obtained in real-time, but in the case of livestock, the voltage may not be constant, so it has the disadvantage of taking care about factors affecting power supply and weather. Therefore, it is judged that the methodology should be selected according to the situation, such as weather or electric power secured at the barn site.

About the experiment in this study, the general dynamic flux chamber experiment is mainly conducted following the EPA's chamber manufacturing specifications, so it is judged that the difference due to volume and related flow rate will not be large. Therefore, the stabilization time of this study could be considered. However, if the size and the flow rate are different, it is considered that the related factors should be additionally considered by referring to the contents of this study.

In addition to the results of this study, if studies such as the difference in stabilization time according to seasons and ambient temperatures and the number of samples that can be represented in the development of emission factors are conducted, it may contribute to improving the reliability of ammonia inventory in the livestock sector.

Author Contributions: All authors contributed to the research presented in this work. Their contributions are presented below. Conceptualization, E.-C.J.; Methodology and writing—original draft preparation, S.K. and, Analysis, Y.H., M.S.I.; Data curation, S.-D.K. All authors have read and agreed to the published version of the manuscript.

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