

## 研究資料 (Research Material)

# A Method for Measuring Fluxes of Greenhouse Gases from Forest Soils

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### Abstract

A method for measuring the flux of greenhouse gases (GHGs) from a forest floor is described. The method follows the non-flow-through closed chamber method in principle and we have used the method all over Japan. The advantages of this method are simple, low-cost, and applicable in remote sites without electricity. The equipment, procedures for installing the chamber and collecting gas samples are described in detail. The paper also gives an analysis of gas samples, a design of the research plot.

**Key words :** greenhouse gases fluxes (GHGs fluxes), carbon dioxide (CO<sub>2</sub>), methane (CH<sub>4</sub>), nitrous oxide (N<sub>2</sub>O), forest soil

## 1. Introduction

Global warming caused by increasing concentrations of greenhouse gases (GHGs) in the atmosphere is of great concern worldwide. The United Nations Framework Convention on Climate Change (UNFCCC) was adopted in 1992 and implemented on 21 March 1994. Countries (Parties) ratifying the convention are required to review anthropogenic emissions and removals of carbon dioxide and other greenhouse gases periodically.

Among GHGs, carbon dioxide (CO<sub>2</sub>), methane (CH<sub>4</sub>), and nitrous oxide (N<sub>2</sub>O) dynamics have close relationships with soil processes in terrestrial ecosystems. In forest ecosystems, many researchers have been measuring these GHGs fluxes from the soil surface. However, present methods employed vary among researchers and countries and there is no international standard. For example, the size and shape of the chamber, the detectors of gases, and the intervals of sampling may differ depending on the purpose of research, accessibility to the field, and cost and convenience of equipment.

In the first stage, it is necessary to accumulate data on annual or monthly GHGs fluxes from various forest soil types in Japan to establish an inventory on GHGs fluxes. However, measuring gas fluxes in forests often have many restrictions, for example, remote field, no electricity, and rough surface conditions. Costly high resolution monitoring system such as automatic flux monitoring would be difficult to install and be unnecessary for our purpose. Simple system is favorable for measuring fluxes under a variety of field conditions. Therefore, we employ

a non-flow-through closed chamber method, which has been used by many researchers for decades. This method is advantageous for determining CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O fluxes with the same sample at low cost. Although fluxes from forest floors are spatially heterogeneous, a relatively wide chamber size would show average flux with small deviation.

In this report a method for monitoring GHGs fluxes from forest soils in Japan is described. And a design of a study field is also described, because the arrangement of chambers is also a factor affecting the average flux.

## 2. Establishment of Study Plot

### 2.1. Plot design

A study plot is 28 m × 28 m (horizontal distance) square plot which is divided into seven 4 m × 4 m quadrats. Slope, vegetation, and soil are favorable as uniform as possible in the plot. Each quadrat is identified by alphabet and number (Fig. 1). A letter from A to G is assigned to each on the column of the quadrat from left to right hand side. Likewise, each row of quadrats is given a number from 1 to 7 in order of top to bottom of the slope, or, north end to south end if the plot is flat. Every corner of the quadrats, each grid, has a pole for identifying the position.

### 2.2. Tree enumeration

Diameter at breast height (dbh) and tree height of trees located in the enumeration plot which corners B2, F2, B6, and F6 quadrat, are measured. The smallest tree

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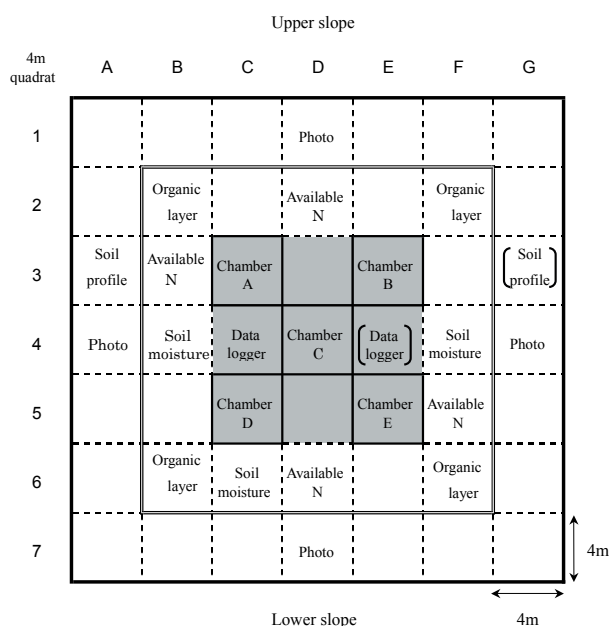


Fig.1 The layout of study plots for gas flux monitoring and the arrangement of equipments and sampling positions.

for the enumeration is 5 cm of dbh. Each tree is given an identification number.

### 2.3. Install gas flux chambers

Five chambers are installed in the quadrats C3, E3, D4, E5, and C5. The method for installing the chamber is described in chapter 3 of this reported in detail.

### 2.4. Install data logger for monitoring micro-climate condition

Before installing sensors and data loggers, the user should master the handling of the equipment. A set of data loggers and sensors is installed in quadrat 4C or 4E. A soil moisture sensor and a soil temperature sensor are installed at a depth of 5 cm from the surface. Sensor cables are protected by hard plastic tubes to protect them from animal bites. The area where the sensors are installed is indicated clearly not to step on it. Data are stored in the data logger every hour.

### 2.5. Soil survey and sampling

#### 2.5.1. Representative soil profile

A standard soil profile is made at a quadrat at the edge of the plot (e.g., G3 or A3). Select a quadrat where soil conditions are similar to those of the chamber. The soil profile is described following either the Soil Survey Manual of Japanese Forest Soils (Forest Soil Division, 1976) or international method (FAO et al., 1998).

#### 2.5.2. Soil sampling from the pit

Soil for chemical analyses is sampled at depths of 0 – 5, 5 – 15, 15 – 30, 30 – 50, 50 – 75, and 75 – 100 cm.

Three 100 mL core samples are also collected from the same sampling depths to be used for measuring physical properties of soils. Chemical and physical properties of the soils are measured in the soil analysis manuals such as “Methods of Soil Analysis Part2” (Page et. al., 1982) and “Methods of Soil Analysis Part1 (Klute, 1986)” .

#### 2.5.3. Soil sampling for available nitrogen analysis

Samples for determining the available nitrogen content of the soil are sampled from quadrat D2, B3, F5, and D6. After taking samples from the forest floor (L and F, or Oi and Oe layers), soil blocks of 25 cm × 25 cm × 5 cm depth are collected. Collected soils are transported to the laboratory under cool condition (5 °C). The available nitrogen of the soil is measured using the incubation method at 25 °C for 4 weeks (Page et. al., 1982).

### 2.6. Organic layer sampling

The weight of the organic layer is measured in autumn before the season of leaf litter fall. A 50 cm × 50 cm frame is set on the forest floor at quadrats B2, F2, B6 and F6 away from standing trees. Leaf litter and detritus (L and F layers, not including the H layer) inside the frame are collected. Branches larger than 1 cm diameter are collected separately. Oven dry weight (70 °C, 48 h) of the organic materials is measured.

## 3. Method for GHGs fluxes measurement

### 3.1. Preparation for installing chambers in the field

#### 3.1.1. Chambers and equipment for installing

Fig. 2 shows the chamber and tools for installing. A small shovel, knife, gardening scissors, and scale are useful. The size and shape of the stainless steel chamber is 40cm diameter and 15cm height with a flat side rim on the top. The cover of the chamber is made by PVC with 3 mm thick. The edge of the cover has a silicone ring with about 1.5 cm in width and 2 mm in thickness. There are two holes with 5 mm on the cover; one for rubber septum and the other for air pressure regulating bag (Fig. 2). Before installing, write numbers from 1 to 8 are written on the inside wall of each chamber with a pen in the laboratory (Fig. 3)

#### 3.1.2. Setting the chambers

The chamber should be placed in a typical location in the study site. Usually this means it should not be placed near trunks, mounds, basins, coarse woody debris or rocks. Put a chamber on the forest floor and cut organic layer and soil using a gardening scissors or a knife along the edge of the chamber (Photo.1). If there are barriers such as big roots or a stone that impede installation of the chamber, it is better to change the position. Insert the

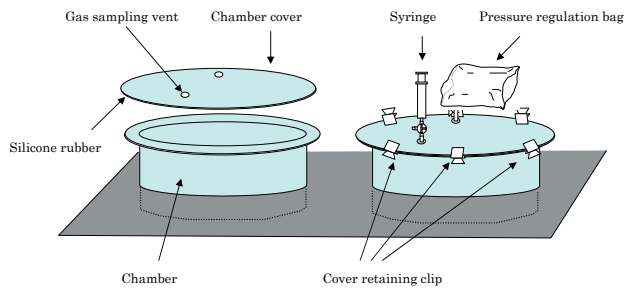


Fig.2 The schematic diagram of the chamber.

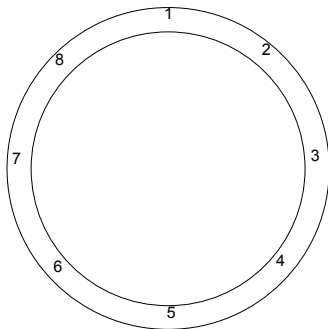


Fig.3 Mark the number on the edge of each chamber.

chamber into soil down to around 3 - 5 cm depth. Measure the angle of the slope degree of the chamber. Confirm that the chamber is fixed tightly on the forest floor without space between the chamber rim and the soil surface. Since the disturbance caused by installing the chamber may affect gas flux, it would be better to wait for a while, usually a month, before starting gas sampling.

If the effects of forest management such as harvesting and thinning on gas fluxes are studied, it would be better to remove the chambers during those operations to be prevented from being damaged. After the operations are finished, re-install them in as close to the same condition

before removal as possible.

### 3.2 Preparation for gas sampling

#### 3.2.1. List of equipment for gas sampling

1. PVC lids for covering chambers.
2. Syringes (two type): A 50 mL syringe is connected with a three-way cock. The syringe connected with a needle is used for gas sampling. The other without a needle is used for putting ambient air into a pressure-regulating bag.
3. Four evacuated vials for one chamber.
4. Timers. A timer that is able to set three different times independently is preferable.
5. An air bag for regulating pressure
6. Eight clips for a chamber
7. A scale for measuring the height of the chamber
8. Rubber septum and disposable needles (for spare parts)
9. A digital thermometer to measure air and soil temperature
10. A field notebook

#### 3.2.2. Preparation of evacuated vials

For collecting gas samples in the field, glass vials are evacuated below 10 Pa by an evacuator (for example, a freeze drier can be used for this purpose) in the laboratory. Seal the vial with a butyl rubber stopper. Butyl rubber is the best choice to get a leak-proof condition. According to our preliminary experiment with a standard gas, leakage of the vial was negligible for several months. Put a tag on the cap of the vial after evacuation to indicate that the vial is evacuated. After sampling, the tag is removed to show the vial is filled with sampled air. The glass vials are safely transferred in an appropriate box.



Photo.1

- A) The installation operation of the chamber. A researcher cut the soil with a gardening scissors along the edge of the chamber for easy insertion of the chamber.
- B) The chamber after the installation.

### 3.3. Gas sampling in the field

#### 3.3.1. Check condition of chamber

Check the condition of the chambers in the field before gas sampling. If a chamber is disturbed, for example, by being moved or tilted, write the conditions of the chamber in the field notebook and re-install it. If twigs and litter are hanging on the rim of the chamber, remove them. Wipe the rim before covering the lid.

#### 3.3.2. Preparation of pressure regulating bag

Connect a disposable syringe to the tube of an air pressure-regulating bag. Evacuate air in the bag to confirm that no leakage occurs in the bag and joint part. Then, inject 160 mL ambient air into the bag with the syringe. Make sure not to contaminate the air with your breath. Turn the cock of the bag to close the bag. Insert the tube of the bag into a hole in the lid.

#### 3.3.3. Set alarm timer and equipment

Set alarm timers at 9, 19, and 39 minutes. Tighten the joint of the syringe and three-way cock again. Place the following items near the chamber: 1) A lid with pressure regulator bag (Do not damage silicone rubber seal), 2) At least 8 clips for fixing the lid to the chamber, 3) Four evacuated sampling vials, 4) A timer.

### 3.4. Gas sampling procedure

The gas should be sampled between 10 a.m. and 3 p.m. for measuring the daily average gas flux in the field. If a sample is taken before or after the hours above, record the sampling time for taking into consideration.

Impact on chambers may cause a serious error in gas flux measurement. Handle the chambers as delicately as possible during sampling.

Turn the cock of the pressure-regulating bag to open. Close a lid before gas sampling (Photo.2). Fix the lid to the chamber using one or two clips as soon as possible. Start the timer to take a gas sample at the time 0 minutes (initial gas sampling).

Plunge the needle of the syringe into the septum of the lid. Turn the three-way cock of the syringe to open. Take a 40 mL air sample from the chamber with the syringe (Photo.2). Turn the three-way cock to close. This procedure should be finished within 10 seconds.

Pull the needle out of the septum, holding the septum with a finger. Clip the lid and the chamber from all directions to seal off air in the chamber. Record the sampling time (year, day, hour, minutes) in the field notebook.

To transfer the gas in the syringe to a glass vial, stick the needle of the syringe into the center of the rubber septum of a vial (Photo.2). Turn the three-way cock to open. Push the handle of the syringe to transfer all the gas

(40 mL) sampled into the vial (30 mL) in order to make the internal pressure of the vial higher than atmospheric pressure. Turn the three-way cock to close and then pull the needle out of the vial. Remove the tag on the vial cap to indicate the vial now contains a sample. The vials should not be placed direct sunlight.

Take gas samples at 10, 20, and 40 minutes after the lid is closed. The timer alarm informs you 1 minute before to start the sampling. Procedures of gas sampling are the same as those for the initial sampling.

After the last gas sampling at 40 minutes, take all clips off to remove the lids from the chamber. Measure heights (mm) of the chamber at 8 marking points to determine the volume of the chamber. The height from the soil surface, the boundary between mineral soil and organic layer, to the edge of the chamber is measured but disturbance of the organic layer should be minimal. If the organic layer is very thick, i.e. thicker than 10 cm, the height from the surface of the organic layer is measured to protect it from disturbance. In this case, it is necessary to determine the volume of the organic layers by physical analysis using a core sampler. Volumes of solid and liquid phases in the organic layer are substituted from the total chamber volume to adjust the actual volume in the chamber.

### 3.5. Other remarks

Do not step on or otherwise disturb the area around the chamber. Compaction of the soil surface around the chamber may cause serious errors in the flux data.

## 4. Gas analysis

The gas concentrations of CO<sub>2</sub> and CH<sub>4</sub> are determined by a gas chromatograph (GC) with a thermal conductivity detector (TCD) and a flame ionization detector (FID). The concentration of N<sub>2</sub>O is determined by GC with an electron capture detector (ECD). The analytical condition of gas chromatograph is shown in Table 1.

For the determination, 5 mL of gas sample is first taken from the glass vial and injected into the GC-TCD-FID for CO<sub>2</sub> and CH<sub>4</sub> analysis. Then, 1 mL of gas sample is taken for N<sub>2</sub>O analysis. This order of analyses, first CO<sub>2</sub> and CH<sub>4</sub>, and then N<sub>2</sub>O, is important to avoid contamination with carrier gas of GC-ECD that contains 5% CH<sub>4</sub>.

## 5. Calculation of flux

Gas flux is calculated by the following equation (Hutchinson and Mosier, 1981; National Astronomical Observatory, 1992),





Photo.2

- A) The chamber is closed by the white lid with pressure regulator bag. The sampling time will be alarmed by the timer, which is placed on the near side in this photograph.  
 B) A researcher is taking a gas sample with the syringe for gas sampling.  
 C) The gas sample is transferred to an evacuated glass vial.

Table 1 Analytical condition of gas chromatograph.

		CO <sub>2</sub> and CH <sub>4</sub>	N <sub>2</sub> O
Column		Unibeads C	Porapak N
Inner Diameter	3 mm		3 mm
	Length	2 m	2 m
Carrier gas		N <sub>2</sub> or He	5% CH <sub>4</sub> in Ar
Temperature	40 mL min <sup>-1</sup>		10 mL min <sup>-1</sup>
	Column	120 °C	50 °C
	Detector	120 °C	340 °C
Sample size		5 mL	1 mL
Other equipment		Autosampler	Pre-cut system is recommendable with a magnesium perchlorate column

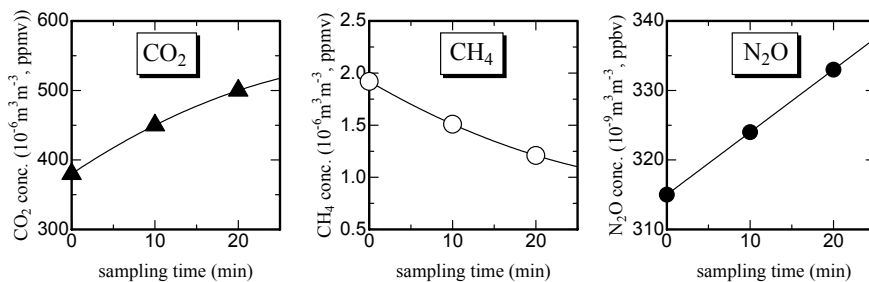


Fig.4 The typical pattern of the changes of GHGs concentrations in the chamber with sampling time.

$$F = \rho \cdot \frac{P}{P_0} \cdot \frac{V}{A \cdot \cos(D)} \cdot \frac{(C_1 - C_0)^2}{t(2C_1 - C_2 - C_0)} \cdot \ln\left(\frac{C_1 - C_0}{C_2 - C_1}\right) \cdot \frac{273}{273 + T} \quad (1)$$

in the chamber.

$$\frac{P}{P_0} = \left(1 - \frac{0.0065z}{T + 0.0065z + 273}\right)^{5.257} \quad (2)$$

where  $F$  is flux ( $\text{kg m}^{-2} \text{s}^{-1}$ ),  $\rho$  is the density of the gas ( $\text{kg m}^{-3}$ , 1.96 for CO<sub>2</sub>, 0.716 for CH<sub>4</sub> and 1.96 for N<sub>2</sub>O at 1013hPa),  $P$  is the atmosphere pressure (hPa),  $P_0$  is the atmosphere pressure at sea level (hPa),  $V$  is volume of chamber ( $\text{m}^3$ ),  $A$  is the area of the chamber ( $0.1256 \text{ m}^2$ ),  $D$  is angle of slope degree of the chamber ( $^\circ$ ),  $t$  is the interval of sampling time (s),  $C_x$  is the concentration of each gas at a time  $x$  ( $\text{m}^3 \text{m}^{-3}$ ),  $T$  is the temperature ( $^\circ\text{C}$ ) in the chamber at gas sampling time and  $z$  is the attitude (m). In this equation,  $V/A$  is equal to the average height of the chamber. Fig.4 shows an example of a gas concentration

## 6. Measuring environmental conditions.

### 6.1. Soil moisture content

Take three soil samples from a depth of between 2.5 and 7.5 cm using a 100 mL or 400 mL cylindrical sampler in the quadrats indicated. Seal the samples and take them back to the laboratory. Measure the oven dry weight (105  $^\circ\text{C}$ , 48 h) of the samples in the laboratory.

### 6.2. Air temperature

Measure the air temperature at 1.5 m above the ground with a thermometer (in 0.1 $^\circ\text{C}$  resolution). Measure the temperature at the beginning and the end of the gas sampling. Record the time that the temperature was

measured. Keep the sensor of the thermometer out of the sunlight.

### 6.3. Data collection from the data logger

Collect temperature and soil moisture data from the logger using a data collector unit. Record 1) soil temperature, 2) output from the soil moisture sensor, and 3) time when data collected, in the field notebook. See the manual of the logger for the procedure to collect the data. After collection, restart the logger according to the manual.

If you have a trouble with the logger (for example, the wire of the sensor breaks), record it and repair as soon as possible.

## References

- FAO, ISRIC and ISSS (1998) World Reference Base for Soil Resources, World Soil Resources Reports **84**, FAO, Rome
- Foest Soil Division (1976) Classification of forest soil in Japan 1975, Bull. Gov. For. Exp. Sta., **280**, 1-28.
- Hutchinson, G.L. and Mosier, A.R. (1981) Improved soil cover method for field measurement of nitrous oxide fluxes, Soil Science Society of American Journal, **45**, 311-316.
- Klute, A. (1986) Methods of Soil Analysis Part1, ASA Inc. and SSSA Inc., Madison
- National Astronomical Observatory (1992) Chronological Scientific Table 1993, Maruzen, Tokyo
- Page A.L., Miller, R.H. and Keeney, D.R. (1982) Methods of Soil Analysis Part2, ASA Inc. and SSSA Inc., Madison

## 森林土壌からの温室効果ガスフラックス測定法

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### 要 旨

我々が日本各地の森林土壌からの温室効果ガスフラックスの測定法として用いている非通気型密閉チャンバー法について示した。この方法の利点は単純、低価格、電源がない遠隔地のサイトでも適用が可能な点である。本報告では測器や器具、チャンバーの設置、ガス採取の手順などの詳細を記述し、サンプルガスの分析や試験地のデザインについても述べた。

**キーワード：** 温室効果ガスフラックス (GHG フラックス)、二酸化炭素 (CO<sub>2</sub>)、メタン (CH<sub>4</sub>)、亜酸化窒素 (N<sub>2</sub>O)、森林土壌

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