Olfactory Measurement Method
Quality Control Manual

Chapter 1 Purpose

Focus !

Purpose

Present various strategies for controlling measurement quality of the olfactory measurement method, and plan for implementation of reliable measurement.

Following the revision to the Offensive Odor Control Law in 1995, regulations based on the "olfactory measurement method," which uses human olfaction as a measurement method, was established, and after a development of laws, odor regulation based on the odor index and odor emission rate (OER) has been fully established. It can be given that the background for this is that in the past, most of the odor sources of complaints were livestock farms and chemicals factories, but have started shifting towards the service industry, such as restaurants, and towards city/life areas, such as housing. As a result, regulations for substance concentrations are not acknowledged as being sufficiently effective, and it has become necessary to respond to offensive odors, which arise from complex odors. When it was established, the olfactory measurement method was confirmed as having an accuracy that compares favorably with instrumental measurement of specified offensive odor substances. However, because there was unfamiliarity with the concept of using human olfaction, it cannot be said that the reliability of the measurement results were widely acknowledged, and to popularize the olfactory measurement method, implementation of quality control and progress in the reliability of measurement results are essential. As the demand for quality assurerance and quality control has been increasing internationally, even in Japan, which has contributed to scientific and technological developments, there is a necessity to actively exercise leadership and plan the establishment of olfactory measurement method that allows for quality control. Consequently, this manual was created with the purpose of planning the implementation of measurements with reliability, by giving various strategies for quality control of the olfactory measurement method.

In the olfactory measurement method, there is the triangular odor bag method (for obtaining the odor index of gas) and the triangular odor flask method (for obtaining the odor index of effluent), but as an initial step, statements regarding quality control in this manual focus on the triangular odor bag method. However, fundamental concepts related to quality control are the same for both methods.

Chapter 2　Overall Framework of Quality Control

Focus !

Basic Framework of Quality Control

① Organization/System for Quality Control
② Quality Control in Measurement
③ Quality Control Using Reference Material

Figure-1 displays the overall framework of quality control that corresponds with the contents of each chapter. The following are explanations of the basic concepts of each topic.

Organization/System for Quality Control

To ensure reliability of olfactory measurement, the development of the organizations and systems that are involved in measurement are necessary, and by establishing a systematic personnel composition, it is possible to clearly divide the roles of each person, and to pinpoint where responsibilities lie. It is also necessary to make efforts to secure the qualifications of people who conduct measurements through periodic education and training. In addition, in olfactory measurement, because the sample is lost when measured, it is not possible to measure the same sample again. Therefore, even after measurement is finished, it is necessary to pay adequate attention in arranging the results and keeping the records so that a series of measurement processes can be accurately comprehended. Details regarding this matter will be discussed in **Chapter 3**.

Quality Control in Implementation of Measurement

Upon implementing measurement, in addition to referring to the measurement manual to curtail error caused by the measurement method to a minimum, create Standard Operating Procedures (SOPs) to be used at each measuring laboratory, and unify operating procedures, including detailed contents that are not stated in measurement manuals. It is also necessary to reduce influencing factors by understanding the points that need to be considered in each operation, from preparing for measurement to calculating results, and to making a checklist. In the end, measurement results are summarized into a report, with the calculation processes of results and their explanations expressed clearly. When tracing archival history of individual samples after measurement, as the results report will fulfill an important role, in addition to recording the details of measurement, establish a storage system. Details will follow in **Chapter 4**.

Quality Control Using Reference Material

As a means for ensuring reliability of measurement results, the evaluation of the measurement results using reference material can be given. Independent quality control can be carried out by conducting periodic measurement using reference material at a measuring laboratory, and comparing them to judgment standards. Moreover, in the future, by using reference material acquainted with values, it will be possible to evaluate the consistency of data between measuring laboratories. Details will follow in **Chapter 5**.

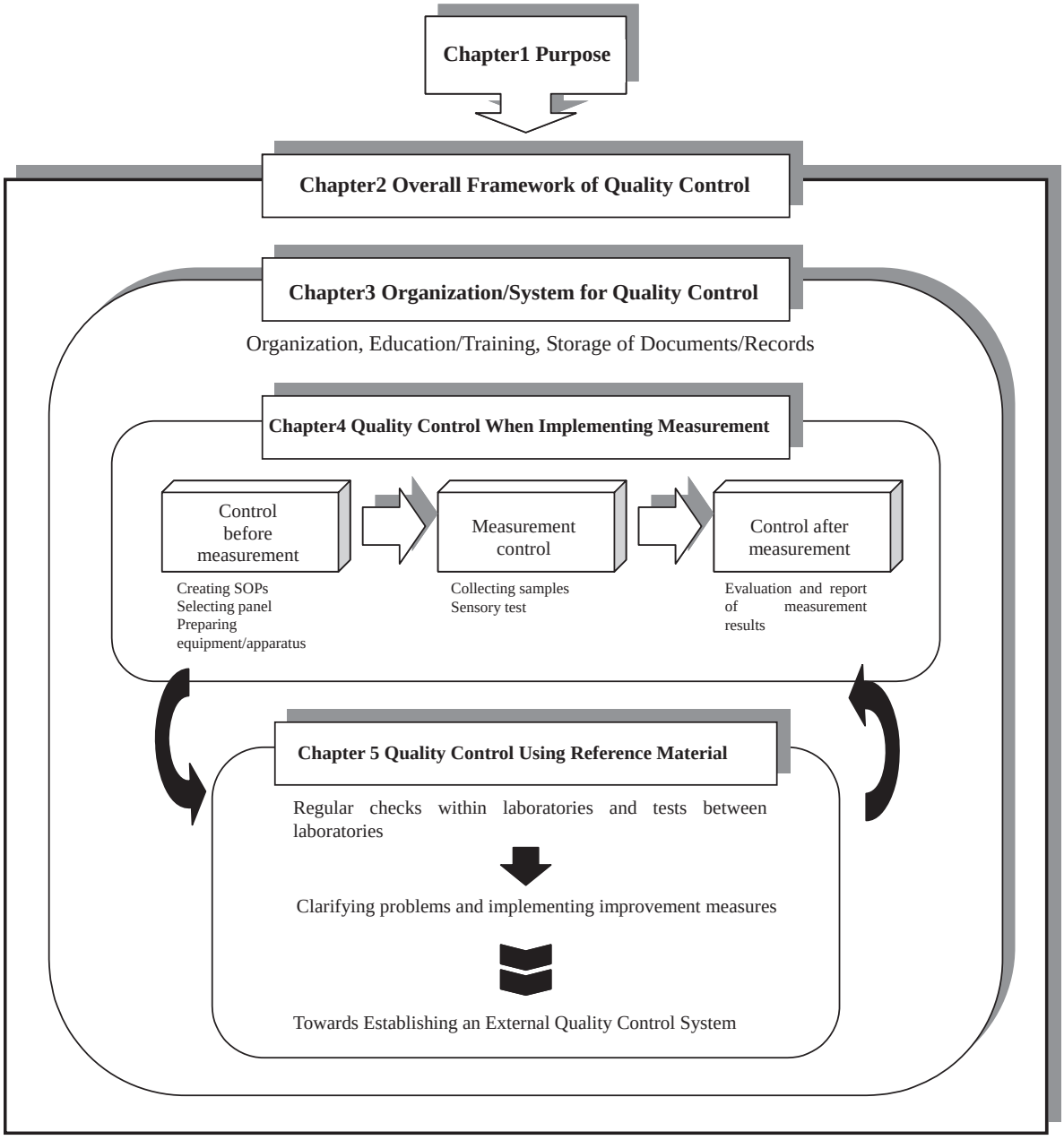


Figure-1 Overall framework of quality control for the olfactory measurement method

Chapter 3　Organization/System for Quality Control

Focus !

Organization/System for Quality Control

① Systematic personnel composition → divide roles, pinpoint responsibility

② Education/training → secure qualifications of person conducting measurements

③ Managing documents/records → understand process up to calculation of results

3.1 Organization

At the measuring laboratories that implement olfactory measurement, it is necessary to divide the roles of the people responsible for implementing olfactory measurement, and to define where responsibilities lie, by clarifying the overall structure of the organization.

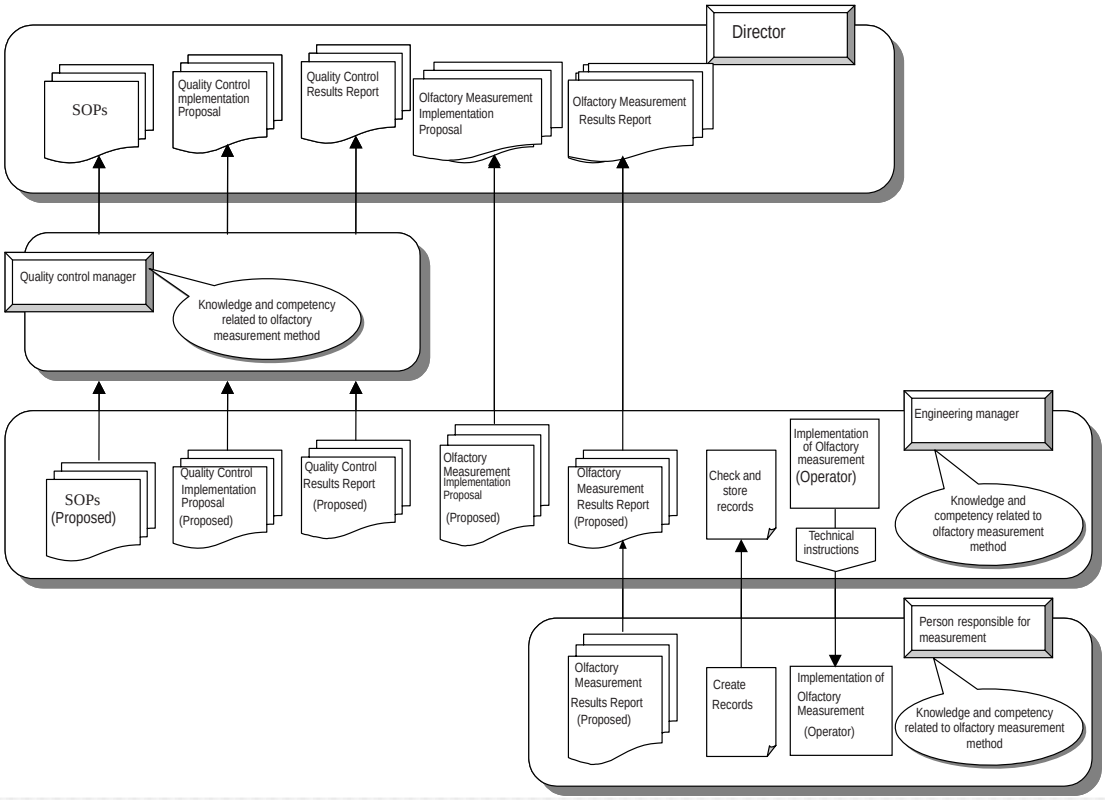
Advice

Example of Organizational Members and Division of Roles

As organizational members, an example such as the one below, where there is a director, a quality control manager, an engineering manager, and a person responsible for measurement, is given. Here, the quality control manger, engineering manager, and person responsible for measurement work closely with quality control, so it is preferable that they have sufficient knowledge and competency in regards to the olfactory measurement method. However, in the case that the person responsible for measurement simply has a supplementary role to that of the person who implements olfactory measurement (operator), these conditions need not apply.

Organization members	Division of roles
Director	Has an overall responsibility in regards to implementation of olfactory measurement. ① Authorizes olfactory measurement implementation ② Reviews and authorizes SOPs proposal, quality control implementation proposal, and quality control results report submitted by the quality control manager ③ Reviews and authorizes olfactory measurement implementation proposal and olfactory measurement results report submitted by the engineering manager ④ Makes improvements of problem areas related to quality control by taking into account the opinions of the quality control manager and engineering manager

Quality control manager	Has sufficient knowledge and competency regarding the olfactory measurement method. Responsible for quality control in regards to olfactory measurement. ① Reviews SOPs proposal, quality control implementation proposal, and quality control results report submitted by the engineering manager, and submits them to the director ② Offers suggestions to the director to improve problem areas related to quality control
Engineering manager	Has sufficient knowledge and competency regarding the olfactory measurement method. Responsible for engineering management of olfactory measurement method. ① Implements olfactory measurement (including preliminary survey and, sampling) ② Gives appropriate technical instructions to the person responsible for measurement ③ Manages and confirms the contents of records, etc. submitted by the person responsible for measurement ④ Makes SOPs proposal, quality control implementation proposal, and quality control results report, and submits them to the quality control manager ⑤ Makes olfactory measurement implementation proposal and submits it to the director ⑥ Reviews olfactory measurement results report submitted by the person responsible for measurement, and submits it to the director ⑦ Offers suggestions to the director to improve problem areas related to quality control
Person responsible for measurement	① Has sufficient knowledge and competency regarding the olfactory measurement method. ② Implements olfactory measurement (including preliminary survey and, sampling) based on instructions from the engineering manager ③ Makes necessary records and submits them to the engineering manager ④ Makes an olfactory measurement results report and submits it to the engineering manager



3.2 Education/Training

To promote quality control effectively, it is necessary to establish opportunities for appropriate education and training (including participation in training at external laboratories), and to plan for improvements in the qualifications and expertise of people who conduct measurement.

3.3 Managing Documents/Records

To be able to accurately understand the series of measurement processes even after measurement has finished, it is necessary to establish a system of managing documents and records. Also, if a problem arises, such as obtaining an anomalous measurement result that is unexpected to be given under the conditions during sampling, there is a necessity to arrange such a system where necessary measures can be taken promptly.

Chapter 4　Quality Control When Implementing Measurement

Focus !

Quality Control When Implementing Measurement

① Create standard operating procedures (SOPs) → unification of procedures, reduce causes of error

② Proper selection and management of the panel

③ Check the points to remember for each operation, create and manage records

- Equipment and Apparatus
- Sampling
- Sensory Test
- Evaluation and report of measurement results

4.1 Creation of SOPs

When implementing measurement, the creation of SOPs will be important. By creating detailed SOPs, it is possible to plan unification of operations at each measuring laboratory, even those that are not stated in the measurement manual, and to reduce causes of error. However, it is necessary to be careful to take case-by-case approaches into consideration at the site. Therefore, while standardizing the measurement manual at actual measuring laboratories, in addition to picking up on other necessary topics, it is recommended to plan out for a uniformity of operating procedures by using an quality control check sheet (Refer to **4.7**).

4.2 Panel

As the olfactory sensitivity of the panel directly affects measurement results, the operator that implements olfactory measurement must select an appropriate panel that does not impair the reliability of the measurement results. It is also preferable that the operator records the selection method of the panel, the results of the olfactory test and the experience of olfactory measurement for each panel member, and saves these records.

4.3 Equipment and Apparatus

4.3.1 Points to Consider

Important points regarding equipment and apparatus are that they should be odorless, and that they can reduce sample loss due to adsorption and prevent contamination by adherence of odorous substances. To keep odorless conditions is very important at every stage of the measurement including equipment and apparatus. **Table-1** summarizes the items that must be confirmed as being odorless, and their countermeasures.

Table-1 Items that must be confirmed as being odorless and their respective countermeasures

Subject	Contents	Countermeasures
Facilities	・ Sensory test room, sample preparation room, waiting room for panel	・ Ventilation, cleaning
Apparatus	・ Odor-free air distributor	・ Exchange activated carbon, filter (absorbent cotton, etc.) ・ Wash diverging tube made of glass
Equipment for sampling	・ Vacuum bottle, sampling bag, etc.	・ Wash vacuum bottle with fragrance-free detergent ・ Wash sampling bag with odor-free air or the subject odor
Equipment for sensory test	・ Syringe, needle, silicone rubber stopper, odor bag	・ Wash with fragrance-free detergent and boil syringe, needle, and silicone rubber stopper ・ Wash odor bag thoroughly with odor-free air
Cabinet to store equipment and apparatus	・ Store equipment and apparatus such as syringes	・ Ensure that they are odorless by activated carbon
Odor-free air	・ Air prepared by aerating through activated carbon tank	・ Management of odor-free air distributor
Panel, operator	・ Cosmetics, hair products, fingers, etc.	・ Education, guidance

When handling equipment and apparatus, it is necessary to pay thorough attention to the following points.

Checkpoints of Equipment and Apparatus

- Wash the vacuum bottle for sampling with fragrance-free detergent and rinse it thoroughly with distilled water. Let it naturally dry in a clean place without any odors and confirm that it is odorless. When storing it, seal it airtight and wrap the sampling opening (branch pipe) with aluminum foil. Before using it, pass odor-free air through it and confirm that the inside of the bottle is odorless.

- Wash the cock of the vacuum bottle and suction bottle for sampling thoroughly with fragrance-free detergent and rinse it with distilled water. Let it dry naturally in a clean place without any odors, and confirm that it is odorless. Do not coat it with grease.

- Use sampling bags made of material (polyester, etc.) that is odorless and has low odor adsorption. Before using it, remove the odor inside the bag by replacing the air in the bag with odor-free air several times, or by continuously letting odor-free air flow into the bag for a given length of time. To confirm that the bag is odorless, fill the inside of the bag with odor-free air, and after leaving it for about one hour, sniff the air inside the bag.

- Use a sampling pump made of highly odorless material that does not adsorb odors easily. In the direct sampling method, pay particular attention when sampling by passing the sample through the pump. Use separate pumps for ambient air samples and exhaust port samples.

- For the tube connected to the sampling bag, use a highly odorless tube made of poly fluoride plastic, but when collecting exhaust gas of high temperature (over 200°C), use a glass tube as necessary. Before using it, wash it well with fragrance-free detergent, and rinse it thoroughly with distilled water. Let nitrogen or odor-free air flow through it to dry it, and confirm that it is odorless.

- Wash the silicone rubber tube well with fragrance-free detergent and rinse it thoroughly with distilled water. Let it dry naturally in a clean place without any odors and confirm that it is odorless. Use one that is as short as necessary.

- Wash the condenser used for removing water and for cooling thoroughly with fragrance-free detergent, and let it dry in a clean place without any odors. Confirm that it is odorless.

- Use a highly odorless pump for supplying air when creating odor-free air. Do not use a pump that has been exposed to odors of high concentrations in the past.

- Wash the activated carbon that is to be used to fill up the odor-free air distributor thoroughly beforehand, and confirm that it is odorless. For details, refer to the "Advice" column below. The frequency for exchanging activated carbon depends on usage conditions; exchange it about once a month if there are many samples, and about once a year if there are not many samples. However, the operator must always pay attention to whether the odor-free air is odorless, and must wash or exchange the activated carbon as necessary.

- As there is the possibility that there is a chemical odor on the filter (absorbent cotton, etc.) used in the odor-free air distributor, wash it thoroughly beforehand, and confirm that it is odorless. For example, after washing it with distilled water, let it naturally dry in a clean room.

- Confirm that there is no odor on the ends of the distributors (diverging tube made of glass, etc.) used in the odor-free air distributor, which can be caused by contact with hands. If there is contamination, remove the odor by washing it thoroughly.

- When not using the odor-free air distributor, prevent odor contamination by wrapping the entire instrument with an odor-free bag.

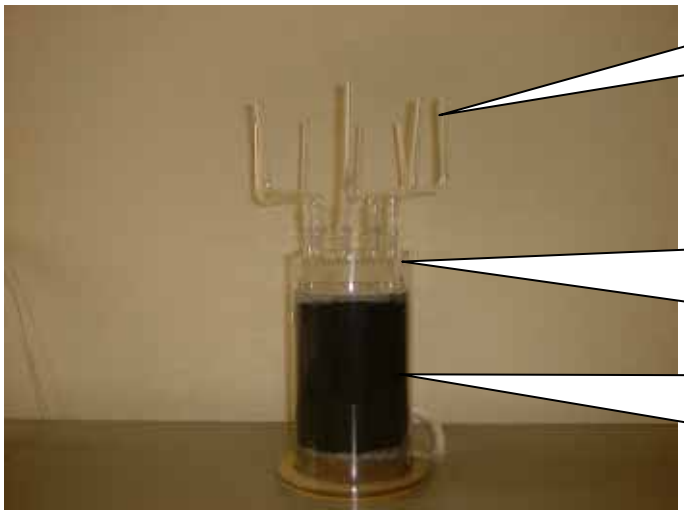
- Use syringes that are odorless. Wash glass syringes thoroughly with fragrance-free detergent, and use it after soaking it in boiled water, letting it dry. Confirm that no odors remain, especially on the sliding part of the syringe. However, as syringe barrels of lure lock glass syringes and plungers of gastight syringes have a risk of breaking, do not boil them or use drying machines. When storing them, prevent them from becoming contaminated by the outside air by putting them in exclusive cases along with the activated carbon, or by wrapping them in aluminum foil. Do not coat them with grease.

- Use odor bags that are highly odorless and have low adsorption and permeability (made of polyester, etc.). Be particularly careful of odor bags for which a long time has passed since they were manufactured, due to a risk of a change in quality or the adsorption of an odor.

- Use sniffing masks that are highly odor-free.

- Wash the silicone rubber stopper well with fragrance-free detergent, and rinse it thoroughly with distilled water. Let it dry naturally in a clean place without any odors, and confirm that it is odorless. If the odor has not been removed sufficiently, boil it, but as silicone rubber stoppers have an inherent odor immediately after being boiled, let it air-dry in a place with no odor for a given length of time (approximately 10 days).

Use the odor-free air distributor after confirming that each part of the activated carbon tank is odorless, as shown in **Photo-1**.



Diverging tube made of glass
Before measurement, wash it with alcohol and remove any sebaceous matters

Filter (absorbent cotton)
Air-dry it, or let it naturally dry after washing it, and confirm that it is odorless before using it.
Be careful that the fine powder of the activated carbon does not disperse and get into the odor bag.

Activated carbon
Use it after washing and drying it in a drying machine. If there is no drying machine, it is preferable to exchange it with a new one.
Use it after removing the fine powder by sifting.

Photo-1　Checkpoints for activated carbon tank

Advice

Example of How to Wash the Activated Carbon

Regardless of whether the activated carbon used in the odor-free air distributor is new or reused, use it for measurement after processing it in the following way, filling it into the activated carbon tank, and confirming that it is odorless by aerating it with a pump.

① Put the activated carbon (granular activated carbon, crushed activated carbon) in a bucket, add distilled water, and leave it for about half a day.

② Remove the oils and fine powder floating on the water surface, and wash the activated carbon several times with distilled water.

③ Drain excess water from the activated carbon rinsed in water, and after drying it using a drying machine without any odors at 100 to 200°C, let it cool down to room temperature.

④ After removing the fine powder on the dried activated carbon by sifting, fill the activated carbon tank with absorbent cotton that has been confirmed as being odorless.

⑤ Use odor-free air only after confirming that it is highly odorless before the test.

◇ Remarks ◇

- The amount of activated carbon decreases approximately 15% due to fine powder being removed during washing and drying. Therefore, prepare a sufficient amount for filling beforehand.
- As a target to determine whether the activated carbon has dried, it is recommended to measure its weight before and after drying.
- Activated carbon for preparing odor-free air that has been prepared in the abovementioned ways is commercially available by some manufacturers.
- When storing activated carbon that has been washed, be careful of contamination by odor adsorption.

4.3.2 Creating and storing records

It is preferable that the operator creates and stores records regarding the following items. By doing so, in addition to improving knowledge appropriate to handling the equipment and apparatus, it becomes possible to grasp the timing of when the equipment and apparatus should be washed, as well as when to exchange consumables. It also becomes possible to accurately know the conditions of the equipment and apparatus during measurement.

1) Equipment

Confirm that the equipment below that is used in olfactory measurement is that prescribed in Notice No. 063 of the Environment Agency, and record their usage, management, and repair conditions.

① Sampling equipment (vacuum bottle, vacuum pump, suction pump, sampling pump, vacuum container, etc.)

② Pump for supplying air

③ Other necessary items

2) Apparatus

Confirm that the apparatus below that is used in olfactory measurement is that prescribed in Notice No. 063 of the Environment Agency, and record their usage conditions, washing methods, and storage conditions.

① Odor-free air distributor (including activated carbon)

② Syringe (syringe made of glass, syringe made of plastic, gastight syringe)

③ Odor bag

④ Sniffing mask

⑤ Silicone rubber stopper

⑥ Other necessary items

4.4 Collecting Samples

4.4.1 Points to Consider

Samples must be properly collected, or otherwise there will be no meaning to the results of the olfactory measurement. With regards to sampling, it is necessary to pay thorough attention to the following points.

Checkpoints for Sampling

- If the site was visited, but not enough information was obtained so that samples could be collected immediately, conduct a preliminary survey. In the preliminary survey, thoroughly comprehend the odor emission conditions (odor emitting processes, fluctuation patterns of environmental odors, etc.) and conditions of the sampling location (selection of location, securing foothold, possibility of using equipment and apparatus, etc.).
- It is recommended that the same person conducts the preliminary survey and collects the samples as well.
- Because time fluctuations are large for ambient air samples, in addition to comprehending the fluctuation pattern as much as possible, collect samples when fluctuations are at their peak.
- When collecting exhaust port samples, implement an appropriate procedure for removing water and dust in the exhaust gas so that they do not cause an influence. For example, use a condenser for removing water, and use a tube plugged with glass wool for removing dust.
- When collecting samples using sampling bags, to avoid losing some of the sample due to adsorption, it is recommended to replace the inside of the bag with the sample several times before collecting a sample.
- When collecting several samples using a sampling pump in the direct sampling method, be careful that there is no influence from the previous sample, by exchanging the part where the sample passes through in the pump.
- When transporting and storing samples, avoid contamination that can be caused by direct sunlight, high temperatures, and miscellaneous odors.

4.4.2 Creating and storing records

It is preferable that the person responsible for sampling creates a record of the following when sampling, and stores it with the measurement results. By doing so, in addition to accurately understanding the conditions at the site, it is possible to properly follow up on archival records of samples after measurement.

1) Preliminary survey

If conducting a preliminary survey, record and store the results.

2) Sampling

The person responsible for sampling makes and stores a record of the conditions during sampling.

4.5 Sensory Test

4.5.1 Points to Consider

In implementing the sensory test, some important factors include ensuring that the odor-free air is odorless, diluting the sample appropriately, and unifying how the panel sniffs odors and their method of answering. Therefore, it is necessary to pay thorough attention to the following points.

Checkpoints for Sensory Test

- In principle, the sensory test is conducted on the same day the sample is collected. If a sensory test on the same day is not possible, conduct a sensory test in the morning of the next day, at the latest.
- If the person who collected the sample is different from the person in charge of the sensory test, be cautious when handing over the sample.
- Ventilate the sensory test room and sample preparation room thoroughly, and maintain a condition of odor-free air constantly. As there are cases in which the existence of odors in a room become undetectable if in the room for a long time, confirm that the air inside the room is odorless by comparing it with the air outside of the room from time to time. Also, when using locations such as a conference room for the sensory test room, ventilate the air sufficiently by prohibiting smoking in the room since the previous day.
- When making odor-free air, let air flow through the odor-free air distributor in the beginning for a while, and use the air in the sensory test after confirming that it is odor-free. For details, refer to the “Advice” column below.
- It is preferable that the temperature of the odor-free air is that of room temperature, but in the case that the temperature rises due to heat from the pump, there is a method of cooling the air by using a cooling water bath and a condenser, and then using the air for measurement.
- Use odor bags after thoroughly washing them. For example, remove any odors inside the bags by replacing the air in the odor bag several times with odor-free air, or by letting odor-free air continuously flow into the odor bag for a given length of time. To determine whether the air inside the odor bags is odorless, put odor-free air into the odor bags, and confirm the presence

or absence of odor. For details, refer to the “Advice” column below.

- Prepare a syringe that is adequate for the capacity of the amount to be injected, and use it after washing it with the sample several times. Confirm that the syringe needle is securely attached, and that the plunger tip of a gastight syringe has not become loose.
- To make sure that the odor of the first diluted sample is not too strong, the operator determines the first dilution ratio carefully. For example, if there are several operators, confirm with other operators, and make the panel respond to the odor intensity. If there are many responses indicating that the value of the odor intensity is over 3, then review the dilution ratio.
- The operator confirms the intensity of the sample at each dilution stage, and not only the sample that is diluted first. The operator also checks whether the dilution procedure is being carried out properly. Be careful when handling complex odors, as there are cases in which the odor characteristics change as dilution progresses.
- If necessary, give guidelines (including a demonstration) to the panel on how to sniff the odor beforehand, and be careful so that the judgment is performed in a unified method.
- If necessary, make the panel sniff odor-free air and a sample diluted moderately before the sensory test, to make them sufficiently aware of the characteristics of sample odor and of the properties of odor-free air.
- If necessary, make the panel give a response to the odor intensity of one set of three odor bags to check the presence or absence of odor.

When making the panel respond to the odor intensity, it is recommended to use an answer sheet such as the one shown in **Figure-2**.

Answer Sheet

Name .....

Out of the three bags, what is the number of the bag with an odor?	What is the extent of the intensity of the odor of the bag that you selected? Circle the applicable number.
	0 Do not know 1 Can be barely detected 2 Weak odor in which the odor can be recognized 3 Can be detected easily 4 Strong odor

Figure-2 Example of Answer Sheet

Advice

Method of Confirming that Odor-free Air Is Odorless

Always confirm that the odor-free air is odorless before using it in judgment. The procedure for confirming that it is odorless is as follows.

(1) Confirm that the air from the diverging tubes made of glass that have been connected to the activated carbon tank are odorless by sniffing each of them directly.

(2) After confirming that the air is odorless in (1), fill the odor-free air into an odor bag that has been thoroughly washed with odor-free air, and sniff the air inside the odor bag immediately after filling it to confirm that it is odorless.

(3) After confirming that the air is odorless in (2), fill the odor-free air into an odor bag that has been thoroughly washed with odor-free air, and after leaving it alone for about 10 minutes, sniff the air inside the odor bag to confirm that it is odorless.

* Remarks *

- It is possible to confirm that the apparatus for supplying odor-free air overall is odorless in (1).
- It is possible to confirm that the odor bag is odorless in (2).
- It is possible to confirm that the sample is odorless when giving it to the panel in (3).
- After being confirmed as odorless in (2), but an odor is detected in (3), some possible reasons could include contamination of the silicone rubber stopper or a manufacturing defect of the odor bag.

4.5.2 Creating and storing records

It is preferable that the operator creates and stores a record of the conditions while the sensory test is being implemented.

4.6 Evaluation and Report of Measurement Results

4.6.1 Points to Consider

Based on the results of the sensory test, a sample’s odor index or odor concentration is calculated, but at this point, confirm that there are not any unnatural results by evaluating the results in a comprehensive way through taking the results of the preliminary survey and sampling results into consideration. If a questionable point arises, review the process up until the derivation of results by tracing back the archival records of the measurement.

4.6.2 Creating and storing records

It is preferable that the operator creates and stores a record when reporting the measurement results.By collectively organizing the series of information, from the preliminary survey to the sampling, sensory test, and calculation of results each time, it becomes possible to plan for a smooth progression of olfactory measurement as a whole. This also allows for a smooth communication of information between each person in charge of operations, and makes it possible to properly follow up on archival records of samples even after measurement.

4.7 Using the Checklist

To conduct olfactory measurement that takes heed of quality control, it is necessary for all people involved in olfactory measurement to thoroughly understand the factors that affect the reliability of measurement results. Consequently, it is recommended to create a checklist that lists the important points concerning quality control, and to proceed while checking each operation. By proceeding with operations in line with the checklist, it is also possible to plan for a unification of operating procedures (Refer to **4.1**). It is recommended to create a checklist by extracting from this list, in response to measurement conditions, the items that are actually necessary.

If there is a case in which there is clearly a problem in quality control from using the checklist, in addition to recording the problem in detail, it is recommended to summarize improvement measures for the problem and its outcomes. By accumulating such information, the implementation of olfactory measurement with higher reliability can be expected.

Chapter 5　Quality Control Using Reference Material

Focus !

Quality Control Using Reference Material

① Reference material…ethyl acetate

② How to prepare reference odor
- Gas cylinder
- Reference gas generator
- Vaporization in air of constant volume
- Disposable can

③ Independent quality control…check within laboratory

④ Collaborative assessment experiment by several measuring laboratories…test between laboratories

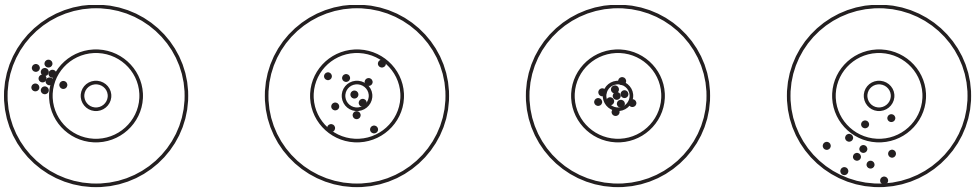
5.1 Overview

An evaluation of accuracy (precision and trueness) using reference material can be given as a means for quality control by the olfactory measurement method. In other words, using reference material for which threshold values are already known, repeated measurement of threshold values are conducted at a measuring laboratory. By comparing the mean value and dispersion of threshold values with judgment standards, an quality valuation of the olfactory measurement method at a measuring laboratory can be conducted. Use values obtained from past collaborative tests by measuring laboratories. Based on the evaluation results, a measuring laboratory can also strive for implementation of olfactory measurement with higher reliability, while understanding the problems that occur in quality control and reviewing methods to improve these problems. Here, as quality control using reference material, one way is to use it at a voluntary means of checking at a measuring laboratory, and another way is to conduct a collaborative experiment between multiple measuring laboratories and make comparisons/reviews among the laboratories. “Precision” refers to the consistency of multiple measurement results, and “trueness” refers to the concordance of the mean value of multiple measurement results with the true value or a reference value (refer to the “Column” below).

Column

What is the difference between “precision,” “trueness,” and “accuracy”?

Now, imagine a situation in which you are facing a target and shooting. You are aiming at the center of the target and firing, but it is not easy to score a bull’s eye. After four people have fired 10 shots each, their bullet holes looked like the illustrations below. From these results, how should the firing skills of the four people be evaluated?



Person A　Person B　Person C　Person D

Here, let’s review from the following two perspectives.

① The way the bullet holes are scattered
② The bullet holes’ proximity to the center of the target

Person A’s bullet holes are clustered closely together, but are off-target overall. Person B’s bullet holes are, on the whole, gathered near the center of the target but are scattered apart. Person C’s bullet holes are clustered closely together, and are also gathered neatly at the center of the target. Person D’s bullet holes are scattered widely apart, and are also off-target. When these kinds of conditions are statistically expressed, the way the bullet holes are scattered is **precision**, and the bullet holes’ proximity to the center is **trueness**. The extent of skill, as a combination of both these aspects, is **accuracy**. The above information represented as a graph is as follows.

	Person A	Person B	Person C	Person D
Precision	○	X	○	X
Trueness	X	○	○	X
Accuracy	△	△	○	X

Even with olfactory measurement, it is possible to conduct an evaluation of precision and trueness with the same concept.

* Caution *
The explanations given here are based on JIS Z 8402-1 (Japanese version of ISO 5725-1).

5.2 Reference Material

Use ethyl acetate as a reference material to conduct quality assessment. When using it simply for checks within the measuring laboratory, it is possible to use reagents sold commercially, but when conducting quality assessment in the framework of external quality control, it is necessary to use reference material with attached values to be able to compare results with other measuring laboratories. However, though a guarantee of reliability by a calibration laboratory is necessary to attach values, it is necessary to review details further in the future, including those of a supply system (refer to **5.6**).

As preparations methods, there is a method of using an ethyl acetate reference gas cylinder, a method of using a reference gas generator, a method of vaporizing ethyl acetate in air with a constant volume, and a method of using disposable cans with reference odor (refer to the “Advice” column below). In preparing with any method, it is necessary to make the concentration of ethyl acetate clear when implementing an quality assessment test.

Reason for selecting reference material

Upon reviewing the results of the "Investigation Studies of Quality Control/Safety management in Olfactory Measurement" established within the Japan Association on Odor Environment as commissioned by the Ministry of the Environment in 2000, ethyl acetate was selected as the reference material necessary in fulfilling the following conditions[*1].

(1) A substance for which creating a reference odor of the fixed concentration is simple, and in addition, the created reference odor is stable.

(2) A substance with relatively superior olfactory properties. In other words, variations in the distribution of olfactory acuity of the panel are small, and the presence of odor can be easily judged.

(3) A substance that is highly safe for the operator and panel, and causes as little health hazards or influence to health as possible.

Advice

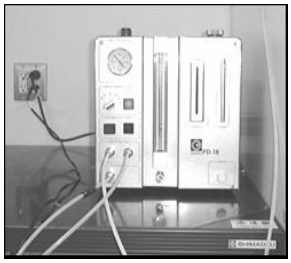
How to Prepare Reference Odor

An outline and points of attention for each method are given below. Based on the results of an examination of the specified concentration and a safety review, there have been favorable findings in all methods [*2].

(1) Method using reference gas cylinder

Obtain a reference gas cylinder from a gas manufacturing company. The concentration of the ethyl acetate should be about 2000 ppm, and the diluent gas nitrogen. The concentration inside the gas cylinder is determined by calibration at the manufacturing company, but its guarantee period is generally less than six months. To ensure that the usage history of the gas cylinder does not affect the odor, it is recommended to tell the gas manufacturing company beforehand that it will be used as a reference gas for olfactory measurement. Use a pressure regulating valve that is odorless.

(2) Method using a reference gas generator



Fill a gas diffusion tube with ethyl acetate (commercially available reagent) and maintain it at a constant temperature to make the amount that is diffused through evaporation constant. If a constant flow of diluent gas (usually nitrogen) flows through it, then gas with a constant concentration can be obtained. Gas concentration is determined by measuring the amount of ethyl acetate that decreases in the gas diffusion tube at constant intervals, obtaining the amount of gas that diffuses per unit of time, and dividing it by the amount of diluent gas. Prior to preparing the ethyl acetate, confirm that the diluent gas is odorless at the opening of the apparatus.

(3) Method of vaporizing ethyl acetate in air of constant volume

After properly injecting odor-free air of a fixed volume into a sampling bag or an odor bag, inject a fixed amount of ethyl acetate (commercially available reagent) using a micro syringe, and let it vaporize.

Or, inject a fixed amount of ethyl acetate into a vacuum bottle using a micro syringe, and after letting it vaporize, connect an odor bag filled with a fixed amount of odor-free air and dilute it so that the overall concentration is uniform. For example, if 80 μL of ethyl acetate is injected into a sampling bag filled with 10 L of odor-free air at 25°C and left to vaporize, the concentration of the ethyl acetate inside the bag is 2000 ppm. As it is necessary to inject small amounts of ethyl acetate using a micro syringe in this method, be extremely cautious when performing this operation.

(4) Method using a disposable can with reference gas

The disposable can with reference gas is one in which ethyl acetate is filled into a disposable can of small capacity at a lower pressure than the gas cylinder, and can be obtained from a gas manufacturing company. The concentration of the ethyl acetate is approximately 2000 ppm, and the diluent gas nitrogen. The concentration inside the disposable can is calibrated by the manufacturing company, and the guarantee period is set by the manufacturing company as well.

The following table summarizes the advantages, disadvantages, and points to remember for each method.

Preparation Method	(1) Gas cylinder	(2) Reference gas generator	(3) Vaporization in constant volume	(4) Disposable can
Advantages	・ Simple to use ・ Concentration of the gas is guaranteed by the gas manufacturing company, stable supply is possible	・ Quality already confirmed by instrumental measurement	・ Low cost	・ Simple to use ・ Concentration of the gas is guaranteed by the gas manufacturing company, stable supply is possible
Disadvantages	Period of concentration guarantee is short (about six months)	・ Takes time for the instrument to become stable	・ Degree of skill has a large influence	・ Period of concentration guarantee is short
Points to Remember	・ Influence of odor of the gas cylinder itself ・ Pressure regulating valve is odorless ・ Change in concentration due to decrease in filling pressure	・ Influence of odor generated from equipment	・ Injection of reagent by micro syringe	・ Influence of odor of the disposable can itself ・ Change in concentration due to decrease in filling pressure

5.3 Implementation of Quality Assessment Test

The method of self-evaluation of measurement quality by a measuring laboratory is described in JIS Q 0033[*3], JIS Z 8402-4[*4], and JIS Z 8402-6 [*5]. The flow of implementing an quality assessment test based on these is as follows (**Figure-3)**. After preparing the reference odor, the measuring laboratory follows the methods of the sensory test of the olfactory measurement method and repeatedly measures the odor index under the repeatability conditions. Afterwards, the common logarithm value of the threshold concentration of ethyl acetate is calculated from concentration of the ethyl acetate that was used as the reference odor and the results of the odor index measurement. However, in actuality, the common logarithm value of the threshold concentration of the ethyl acetate is calculated by calculating the threshold value (common

logarithm value of the dilution ratio related to the threshold value) of the panel and subtracting it from the common logarithm value of the threshold value of ethyl acetate. Implement these operations in each measurement, and use it in the following evaluation.

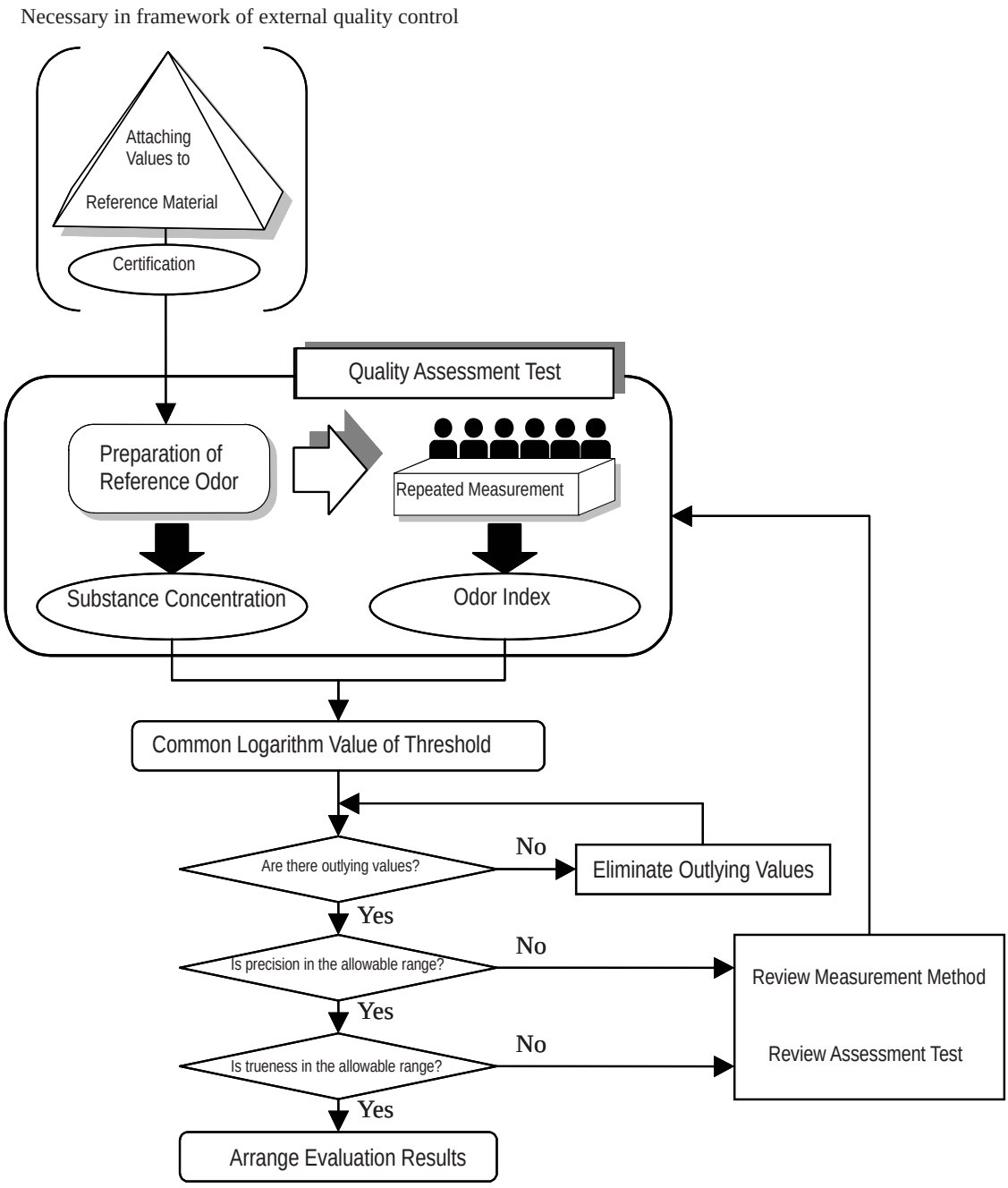


Figure-3 Outline of Quality Assessment Method Using Reference Material

5.4 Evaluation of Results

In regards to the obtained results, conduct a statistical test first to make sure that there are no outlying values. If there is an outlying value, conduct a precision check after eliminating the value. If there is no significant statistical difference with the repeatability standard deviation (a known value determined by collaborative tests) that corresponds to the measurement method, proceed to the trueness check. If there is no significant statistical difference with the reference values (known value determined by collaborative tests) in the trueness check, then it can be determined that the measurement quality (accuracy) at the measuring laboratory is within an allowable range. If it is determined that there is a significant statistical difference in the precision or trueness checks, first conduct a review to determine if there were any problems in implementation of the quality assessment test, and determine whether it is necessary to conduct the test again. If it is determined that there were not any problems in implementation of the quality assessment test, as it is likely that there was a problem of quality control in implementation of olfactory measurement, in addition to determining the cause, it is necessary to review countermeasures for improvement. Here, a checklist and form for recording quality improvement in regards to quality control should be actively used. (Refer to **4.7**).

About Judgment Standard Values for Quality Assessment

Based on the results of cross-check tests using ethyl acetate as conducted by seven measuring laboratories in "Investigation Studies of Quality Control/Safety management in Olfactory Measurement" established within the Japan Association on Odor Environment as commissioned by the Ministry of the Environment in 2000 and 2001, the following mean values for the threshold value, repeatability standard deviation values, and reproducibility standard deviation values were obtained[*6*7]. All values are expressed as the common logarithm value of threshold values.

Sensory test method	Mean value	Repeatability standard deviation	Reproducibility standard deviation
Ambient air sample (implemented in 2001)	-0.10 (0.79 ppm)	0.13	0.24
Exhaust port sample (implemented in 2000)	-0.26（0.56 ppm）	0.17	0.22

As judgment standard values for quality assessment, these values are used as reference values for the time being, but in the future, it is necessary to increase reliability by accumulating suitable data.

The Target for Odor Index of Reference Odor

If the odor index is repeatedly measured with repeatability conditions by the exhaust port sample method using ethyl acetate of a concentration of 2000 ppm, it would be preferable if the standard deviation and mean value of the measurement results fulfill the conditions below, provided that n is the number of repeated measurements.

	n=3	n=4	n=5
Standard deviation	Below 3.0	Below 2.8	Below 2.7
Mean value of odor index	35.5±2.0	35.5±1.7	35.5±1.5

5.5 Frequency of Quality Assessment Tests

To determine the frequency with which each measuring laboratory should conduct quality assessment tests using reference material, it is necessary to decide after comprehensively judging the frequency of olfactory measurement, changes in panel structure, experience of the operator, cost, etc., but it is recommended to use the following as targets for frequency and accumulate data, and based on these results, to review subsequent frequencies for implementation. However, it is necessary to take the actual conditions of measuring laboratories into consideration, and correspond flexibly.

❍ Repeated checks under repeatability conditions

As stated in this chapter, precision and trueness are evaluated based on repeated measurements of reference odor. Fundamentally, repeated measurement is done several times in one year, at least four repetitions in one session. Once a year is acceptable, however, for laboratories with low measurement frequency. In the beginning, try to accumulate as much data as possible and grasp the fluctuations. It is recommended to refer to the values given above as targets for the odor index of reference odor.

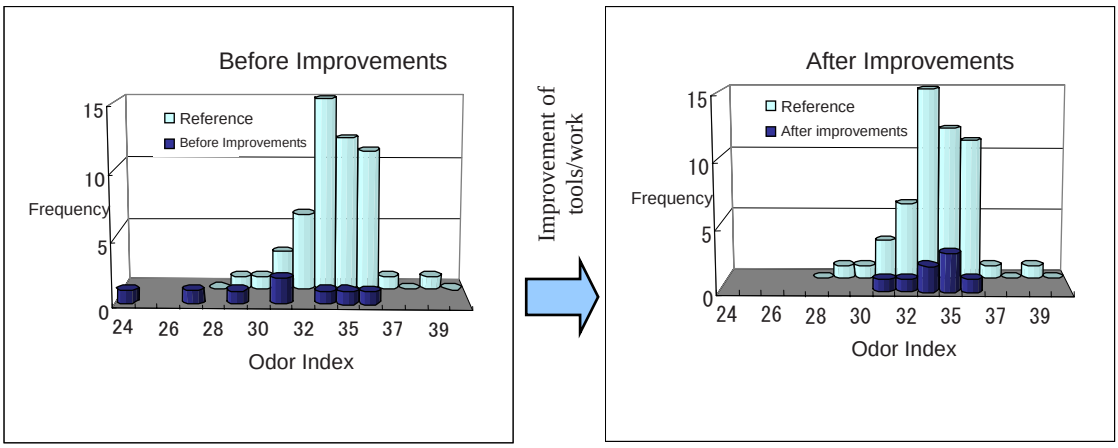
❍ Periodic Checks

These are not repeated measurements, but periodic measurements carried out once at one time. Fundamentally, these checks are conducted once per 30 samples or once a month, whichever one has more frequency. At laboratories that carry out measurement in low frequencies, where the measuring intervals exceed one month, however, checks are carried out in the same frequency as measurements. It is recommended to laboratories that measure at least eight samples a day to perform a total of two checks on the checking day, once first thing in the morning, and another as the last thing in the afternoon. If using ethyl acetate of a concentration of 2000 ppm, the goal is to see whether the odor index can fall within a range of 35.5±3.5.

Column

Example of Implementing Internal Quality Control

In 2001, reference odors were distributed to a total of seven laboratories, including local governments and private analyzing laboratories, and in addition to having them measure the odor index once per odor using the measurement method for an exhaust port sample, instruments used for the sensory test as well as detailed procedures of the sensory test were surveyed. Afterwards, the factors that were thought to have caused influence on the measurement results were conveyed as checkpoints to each laboratory (feedback), and after improvements were made to the equipment and apparatus used and the work contents, they were made to measure the same sample again. The distribution of the odor index measurement results of the seven laboratories before and after the improvements are shown in the diagrams below. Here, reference refers to the target of the distribution when properly measured. By conducting such internal quality control, and improving the influencing factors at each laboratory, the odor index measurement results were also improved.



5.6 Towards Establishing an External Quality Control System

In order for the quality assessment test to be widely used at each measuring laboratory, it is necessary to use a reference material that has secured a quality over a certain level. That is, it is necessary to compare data between measuring laboratories by establishing a supply system of a reference material that has been certified by a calibration laboratory. In the measurement sector, such a system based on the Measurement Law, the Japan Calibration Service System (JCSS), is positioned as follows.

① The Minister of Economy, Trade and Industry has specified a national measurement standard (primary standard)

② The supplying laboratory (designated calibration laboratory) designated by the Minister of Economy, Trade and Industry supplies primary standard

③ The accredited laboratory specified by the Minister of Economy, Trade, and Industry calibrates gauges and attaches values to reference material

The testing/calibration laboratory accreditation system that is based on the Measurement Law is a system in which testing laboratories are accredited by, calibration laboratories that are suited to the ISO/IEC Guide 58 (JIS Z 9358[*8]), based on the ISO/IEC 17025 (JIS Q 17025[*9]). In regards to chemical substances, the Chemicals Evaluation and Research Institute is specified as a calibration laboratory involved with reference gases and reference solutions, and implements concentration reliability tests of reference material that are supplied to general users by accredited laboratories. An overview of such a supply system of reference material is shown in **Figure-4**. If it becomes possible to handle reference material of the olfactory measurement method in the same way as chemical substances, it will probably become necessary to establish a supply system of reference material in the future.

By establishing a supply system of reference material, in addition to independent quality control at each measuring laboratory, applicability to situations such as the following may become possible.

① External audit of a measuring laboratory by a third party laboratory

② Crosschecks during registration screening or those implemented periodically after registration as in the system of accredited laboratories for odor measurement of the Japan Association on Odor Environment

③ Quality assessment collaborative test associated with improvements of the olfactory measurement method or the development of a new measuring method

④ Collaborative tests as voluntary approaches by measuring laboratories to improve measurement quality

In any of the cases above, a third party laboratory will become necessary for substantive execution of quality assessment and for coordinating the implementing laboratories, and in addition to a detailed review of external quality control methods, it is necessary to continue discussing the establishment of the evaluation system and how each measuring laboratory should be involved.

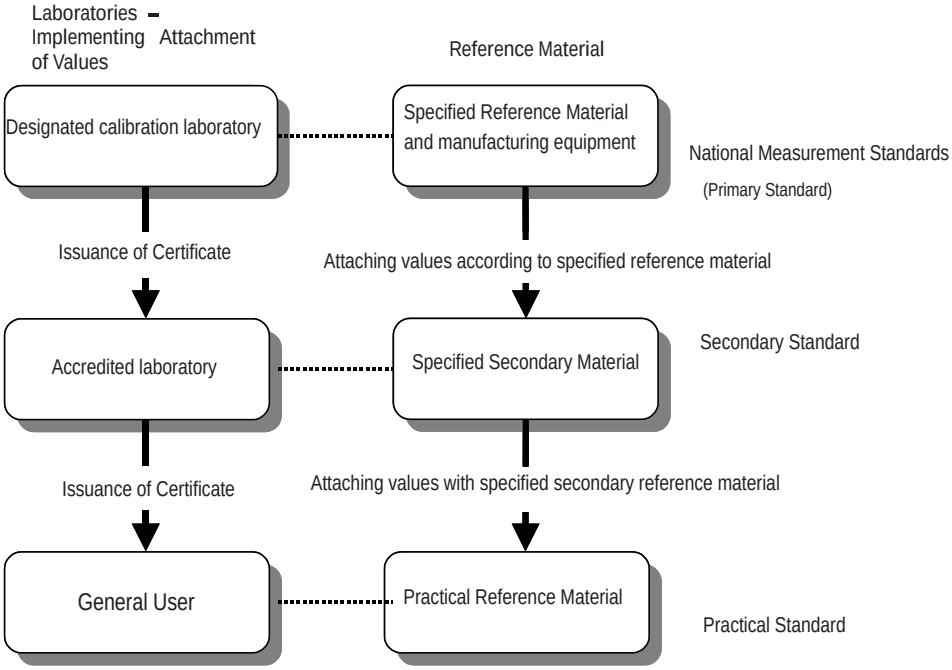


Figure-4 Overview of supply system of reference material