

Guidelines on Quality Assurance/Quality Control for The Environmental Measurements of Dioxins

November 14, 2000
The Environment Agency

These Guidelines set forth measures to be voluntarily taken by laboratories responsible for environmental measurement of dioxins for the realization of appropriate quality control in such measurement activities.

Part 1 General Matters

Chapter 1 Quality Control System

1. Organization

A laboratory that performs environmental measurement of dioxins shall appoint a general supervisor, a quality controller, a technical manager and a measurement technician as detailed below to ensure the proper management of its quality control system.

(1) General supervisor

The general supervisor shall assume responsibility for the entire operation of environmental measurement of dioxins. The general supervisor shall appoint a quality controller, a technical manager and a measurement technician as described in (2), (3) and (4) respectively, prepare a document on the organization containing the names of persons he has appointed, tasks to which they are assigned and their experience in performing their assigned and related tasks (period during which they were assigned to such tasks, record of training they have received, etc.), and make a structural chart of the organization. In addition, the general supervisor shall examine the draft of standard operating procedures as described in 1 of Chapter 2, the draft of a plan for quality assurance and quality control as described in 1 of Chapter 3 and the draft of a report on the results of quality assurance and quality control as described in 2 of Chapter 3 submitted by the quality controller, and approve these drafts.

The quality controller shall be appointed from among persons other than the technical manager or the measurement technician.

(2) Quality controller

A person having the abundant knowledge and superior ability required for quality control in environmental measurement of dioxins shall be appointed as quality controller. The quality controller shall assume responsibility for quality control in environmental measurement of dioxins, examine the draft of standard operating procedures as described in 1 of Chapter 2, the draft of a plan for quality assurance and quality control as described in 1 of Chapter 3 and the draft of a report on the results of quality assurance and quality control as described in 2 of Chapter 3 submitted by the technical manager, and shall submit these drafts to the general supervisor. In addition, the quality controller shall conduct internal audits as described in 3 of this Chapter.

(3) Technical manager

A person having the abundant knowledge and superior ability required for environmental measurement of dioxins shall be appointed as technical manager. The technical manager shall assume responsibility for technical management of environmental measurement of dioxins, give technical instructions concerning the performance of tasks by the measurement technician, and check and retain the contents of records submitted by the measurement technician.

In addition, the technical manager shall prepare the draft of standard operating procedures as described in 1 of Chapter 2, the draft of a plan for quality assurance and quality control as described in 1 of Chapter 3 and the draft of a report on the results of quality assurance and quality control as described in 2 of Chapter 3, and shall submit these drafts to the quality controller.

(4) Measurement technician

A person who has received training in collection of samples, pretreatment of samples, and/or

in measurement by a gas chromatograph mass spectrometer, and who is capable of properly performing these tasks shall be appointed as measurement technician. The measurement technician shall, under the provisions of these Guidelines, prepare and keep in order necessary records, and submit them to the technical manager.

2. Procedures to deal with any operations improperly performed

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The technical manager shall prepare the draft of a document containing the procedures for checking records submitted by the measurement technician and the procedures for dealing with problems that shall be required in cases where the technical manager recognizes any quality control problems while checking the records (hereinafter referred to as "the procedures for dealing with QC problems), and shall submit the draft to the quality controller.

The quality controller shall examine the draft of the procedures for dealing with QC problems and submit the draft to the general supervisor after making modifications as required through consultation with the technical manager.

The general supervisor shall examine the draft of the procedures for dealing with QC problems submitted by the quality controller, give instructions to make modifications as required, and approve the modified draft of the procedures for dealing with QC problems.

In cases where the technical manager recognizes any operations that fail to comply with the quality control requirements specified in these Guidelines, the technical manager shall take proper measures in accordance with the procedures for dealing with QC problems.

3. Internal audit

The quality controller shall conduct audit to ensure that quality assurance and quality control for environmental measurement of dioxins are properly implemented, put the results in the form of a document (hereinafter referred to as "internal audit report"), and submit the results to the general supervisor. Provided that no specific changes have been made to the facilities or equipment in use, or the technical manager has not been replaced by another, audit shall be conducted more than once a year, and if any changes (except minor changes) have been made to the facilities or equipment, or if the technical manager has been replaced, audit shall be conducted at a time when regular measurement is resumed after such changes.

If required, based upon the submitted internal audit report, the general supervisor shall give the technical manager written instructions for quality improvements (hereinafter referred to as "quality improvement instructions").

The technical manager shall prepare the draft of a document containing the measures for implementing quality improvements (hereinafter referred to as "the document for implementing quality improvements") in accordance with the quality improvement instructions, and submit it to the quality controller.

The quality controller shall examine the draft document for implementing quality improvements, and submit the draft document to the general supervisor after making modifications as required through consultation with the technical manager.

The general supervisor shall examine the draft document for implementing quality improvements submitted by the quality controller, give instructions to make modifications as required, and approve the revised draft document for implementing quality improvements.

The technical manager shall take proper measures to improve operations in accordance with the document for implementing quality improvements.

4. Training, etc.

In cases where the general supervisor deems it necessary for the quality controller, the technical manager and the measurement technician to perform their duties, he shall provide training for them (including training provided by external organizations and participation in technical proficiency tests and inter-laboratory comparison tests). When the general supervisor has provided training, he shall require the persons who have received training to submit reports on the contents, period and results of training provided, keep in order and retain these reports,

and add their contents to the document described in 1 (1).

5. Document control

The general supervisor shall prepare a document specifying the procedure for drawing up the documents prescribed by these Guidelines and the procedure for maintaining and controlling these documents (hereinafter referred to as "the procedure for preparing, maintaining and controlling documents/records"), and give instructions to the quality controller, the technical manager and the measurement technician so that these documents/records can be properly prepared, maintained and controlled in accordance with the procedure.

These documents/records shall contain their respective creation dates. For documents listed in 1 of Attached Table 2, the latest editions shall be maintained and controlled, and the previous editions shall be retained for five years as a general rule. Documents listed in 2 of Attached Table 2 and records listed in 3 of the said Table shall be retained for five years as a general rule.

For data prepared by personal computers and stored onto electronic storage media, backup copies shall be produced, and necessary measures shall be taken so that the data cannot be overwritten.

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6. Allotment of a specific task to another organization

In cases where a task such as collecting samples has been allotted to and will be performed by another organization, the details of the allotted task and relations of accountability shall be clarified and contained in the plan for quality assurance and quality control as described in 1 of Chapter 3. In cases where an order for one of the tasks, such as collecting samples, is placed with another organization under subcontract agreement, the details of the ordered task and the organization that receives the order shall be described, and necessary measures shall be taken to ensure that the requirements specified in these Guidelines are implemented.

Chapter 2 Common matters regarding quality assurance and quality control

1. Standard operating procedures

The technical manager shall prepare the draft of standard operating procedures for the tasks performed by the organization concerned among tasks ranging from the management of reagents and collection of samples to the submission of a result report in accordance with the requirements specified in the measuring procedures (procedures prescribed by Prime Minister's Office Ordinance No. 67 of 1999, Environment Agency Notification No. 68 of 1999 and Ministry of Environment Notification No. 80 of 2004 (see Attached Table 1), etc. The same shall apply to the terms used hereunder.) and the requirements contained in Part 2, and submit the draft to the quality controller.

The quality controller shall examine the draft of standard operating procedures, and submit the draft to the general supervisor after making modifications as required through consultation with the technical manager.

The general supervisor shall examine the draft of standard operating procedures submitted by the quality controller, give instructions to make modifications as required, and approve the revised draft of standard operating procedures.

2. Task progress management

After the task of measuring dioxins has been commenced, the technical manager shall comprehend the status of task progress based upon reports made by the measurement technician, make efforts to properly manage the progress, and draw up a record on the progress status.

Chapter 3 A plan for quality assurance and quality control and a report on the results

1. A plan for quality assurance and quality control

The technical manager shall make the draft of a plan for quality assurance and quality control as shown in Attachment 1 (hereinafter referred to as "draft plan" in connection with a series of

operations for environmental measurement of dioxins his organization performs for research services accepted from individual clients.

The quality controller shall submit the draft plan to the general supervisor after making modifications as required through consultation with the technical manager.

The general supervisor shall examine the draft plan submitted by the quality controller, give instructions to make modifications as required, and approve the revised draft of the plan for quality assurance and quality control.

2. A report on the results of quality assurance and quality control

The technical manager shall draw up the draft of a report on the results of quality assurance and quality control as shown in Attachment 2 (hereinafter referred to as "result report") in connection with operations for environmental measurement of dioxins performed in accordance with the plan for quality assurance and quality control as described in 1, and submit the draft to the quality controller.

The quality controller shall examine the draft of the result report, and consult with the technical manager on modifications to be made to the draft if the report does not contain all information required or if it has any other problems. If it has any quality control problems, the quality controller shall consult with the technical manager on measures to be taken in accordance with the procedures (hereinafter referred to as "QA/QC") for dealing with QC problems described in 2 of Chapter 1 and on modifications to be made to the draft of the result report in accordance with the procedures. The quality controller shall submit the draft of the result report that has been modified as required to the general supervisor.

The general supervisor shall examine the draft of the result report submitted by the quality controller, give instructions to make modifications as required, and approve the revised draft of the result report.

In cases where modifications have been made to the original draft, the quality controller shall record the process leading to such modifications.

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Part 2 Particulars

Chapter 1 Management of reagents, instruments, apparatuses and facilities

The measurement technician shall make and keep in order records, etc. on the following items, and submit them to the technical manager. The technical manager shall check the contents of the submitted records, etc., and retain them.

1. Reagents

The measurement technician shall classify reagents to be used according to their applications, check to make sure that they are prescribed by the measuring procedures, and record their manufacturers, product names, lot numbers, purchase dates, purchased quantities, dates on which their bottles were opened, expiration dates, storage methods, etc. For reagents that have been refined, cleaned or subjected to other types of preparation, the measurement technician shall record the person who has performed preparation work, the date on which it was performed and the details of the work.

2. Standard substance (solution)

For standard substance (solution), in addition to all the information specified in 1 that must be recorded, the date on which it was used and the amount used shall be recorded. In cases where standard solution has been purchased, its concentration at the time of purchase shall be recorded (if more than one kind of standard solution has been purchased, the concentration of each kind shall be recorded).

3. Instruments

The measurement technician shall classify instruments to be used according to their applications, check to make sure that they are the same instruments as prescribed in the measuring procedure, and record their manufacturers, product names, any treatment they have

been subjected to, including cleaning, (the name of the person who performed the work, the date on which the work was performed and the details of the work), and storage methods. The measurement technician shall separate instruments for measuring high-concentration samples from those for measuring low-concentration samples, take a necessary measure to clearly identify the two different types of instruments, and record the details of the measure taken.

4. Apparatuses

The measurement technician shall classify apparatuses to be used according to their applications, check to make sure that they are the same apparatuses as prescribed in the measuring procedure, and record their manufacturers, product names, calibration status and how they are maintained in daily measurement activities. In cases where apparatuses have been repaired, the measurement technician shall retain repair slips, and record the conditions about repairs, etc.

5. Facilities

The measurement technician shall draw up a document that allows evaluation of operating conditions to evaluate operating conditions under which a series of operations are performed after the delivery of samples. Specifically regarding operating conditions under which samples are subjected to pretreatment or measurement with a gas chromatograph mass spectrometer, he shall describe care used from the viewpoint of quality control.

Chapter 2 Collection of samples

1. Sampling plan

(1) Preliminary survey

The technical manager shall review the need for a preliminary survey, and if he recognizes the need, he shall require the measurement technician to submit a preliminary survey plan, make modifications to the plan as required, and instruct the measurement technician to conduct the preliminary survey.

The measurement technician shall conduct the preliminary survey according to the plan, record the results of the survey, submit them to the technical manager, and feed back the results of the preliminary survey to the preparation of a sample collection plan described in (2).

The technical manager shall check the content of the record submitted, and retain the record.

(2) Sample collection plan

The measurement technician shall make the draft of a sample collection plan, and submit it to the technical manager.

The technical manager shall make modifications to the draft as required, and then describe it in the draft of a plan for quality assurance and quality control described in 1 of Chapter 3 in Part 1 as a sample collection plan.

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2. Making a decision whether to perform sample collection

The measurement technician shall decide whether to perform sample collection based upon the weather of the previous and current day and other conditions (for certain items, weather conditions of a few days earlier shall be taken into consideration), inform the technical manager of his decision, and obtain the approval of the technical manager. If he has made a positive decision, he shall perform sample collection, and if he has made a negative decision, he shall record the process leading to the negative decision.

3. Record of sample collection

The measurement technician shall perform sample collection according to the sample collection plan, make and keep in order the records listed below, and submit these records to the technical manager. The technical manager shall check the contents of records submitted, and retain the records.

(1) Common matters to be recorded

Names of samples

- Names of collectors
- Date of collection
- Names of sampling sites
- Latitude and longitude of sampling sites obtained by means of the global positioning system (GPS), etc.
- Maps of sampling sites/places and description of their conditions
- Decisions made whether to perform sample collection
- Weather conditions during the collection of samples
- Photographs of sampling activity (two types of photographs; a distant view of the activity showing the surrounding conditions, and a close-range view of the activity showing the sampling conditions. However, photographs shall not be required in cases where it is impossible to take photographs.)
- Matters that are likely to affect samples, including sources in and around sampling sites
- Amount of samples collected
- Means of transportation after the collection of samples

(2) Specific matters for individual measurement items

The following matters shall be specified for each of the individual measurement items in Attachments 3-8.

- Sampling instruments and apparatuses and reagents used
- Sampling activity
- Sample containers

4. Travel blank test and dual measurement

The measurement technician who collects samples shall perform operations for a travel blank test and collect samples for a dual measurement at the time of sampling in accordance with the procedure prescribed in the measuring procedures, and record the conditions of such operations and sample collection.

Chapter 3 Pretreatment of samples

1. Sample pretreatment plan

The measurement technician shall draw up the draft of a sample pretreatment plan, and submit the draft to the technical manager.

After making modifications to the submitted draft as required, the technical manager shall describe it in the draft of the plan for quality assurance and quality control as described in 1 of Chapter 3 in Part 1 as a sample pretreatment plan.

2. Common matters regarding sample pretreatment

The measurement technician who performs sample pretreatment shall perform the following activities according to the sample pretreatment plan, put in order the records he has produced, and submit these records to the technical manager. The technical manager shall check the contents of the submitted records, and retain the records.

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(1) Acceptance inspection of samples

He shall perform an acceptance inspection to check the conditions of samples when the samples collected are delivered to the laboratory, and make a record of the following.

- Date of sample delivery and date of acceptance inspection
- Person who performed the acceptance inspection
- Means and conditions of sample delivery
- Types and sizes of sample containers
- Properties of samples
- Other matters noteworthy to report

(2) Storage and management of samples until extraction operation

For samples that have been subjected to the acceptance inspection, he shall make a record of the following, and be in charge of proper storage and management until he carries out an

extraction operation described in (3).

Control numbers of samples

Sample storage and management places, methods and periods

(3) Extraction from samples

He shall perform an extraction operation on samples, make a record of the following, and make sure that the operation has been performed in accordance with the procedure and under the conditions specified in the measuring procedure. In cases where he has taken a special measure under special circumstances, he shall record the reason, details of the measure and its validity (results of comparative study, documents quoted, etc.).

He shall also make a list of samples he handled at about the same time as standard substance to determine the presence of contamination by other samples during the extraction operation.

Person who performed the operation

Date of operation

Properties and quantity of samples used for extraction

Instruments used for extraction, their cleaning status, and the condition of their storage until use

Procedure for the extraction operation and conditions under which it was performed (Type and amount of solvent, extraction time)

Type and amount of cleanup spike added and the time it was added

(4) Clean up of extract from samples

He shall perform a cleanup operation on the extract from samples, make a record of the following, and make sure that the operation has been performed according to the procedure and under the conditions prescribed in the measuring procedures.

In cases where he has taken special measures under special circumstances, he shall describe the reason, details of the measures and their validity (results of comparative study, documents quoted, etc.).

He shall also make a list of samples he handled at about the same time for future reference to determine the presence of contamination by other samples during the cleanup operation.

Person who performed the operation

Date of operation

Amount of extract from samples used for the operation

Operating procedure and conditions

Type of reagent used

A. Sulfuric acid treatment - in the case of silica gel column chromatographic operation

(a) Sulfuric acid treatment

-Amount of hexane used

-Amount of sulfuric acid added and the number of additions made

(b) Silica gel column chromatographic operation

-Material/product name of silica gel, activation conditions and the amount filled

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-Type of solvent used for elution and the amount of eluted liquid

B. In the case of multi-layer silica gel column chromatographic operation

-Material/product name of silica gel, activation conditions and the amount filled

-Type of solvent used for elution and the amount of eluted liquid

C. In the case of alumina column chromatographic operation

-Material/product name of alumina, activation conditions and the amount filled

-Type of solvent used for elution and the amount of eluted liquid

-Results of fractionation test

D. In the case of high-performance liquid chromatographic operation

-Equipment (the name of manufacturer and model name)

-Material/product name of separation column

-Separation conditions (type of eluent, temperature, flow rate of eluent, time required for separation)

-Blank value

-Order of injection (injection list)

E. In the case of activated-charcoal column chromatographic operation

-Material/product name of activated-charcoal, activation conditions and the amount filled

-Type of solvent used for elution and the amount of eluted liquid

-Results of fractionation test

3. Specific matters for individual measurement items regarding sample pretreatment

Specific matters for individual measurement items regarding sample pretreatment shall be specified as required in Attachments 3-8.

4. Preparation of samples to be measured along with test samples

The measurement technician shall perform necessary pretreatment on the samples listed below to make sure that quality is ensured in connection with measurement of dioxins, prepare them as samples subjected to measurement described in 5 of Chapter 4, record the name of the measurement technician who prepared the samples, the date of preparation and the summary of preparatory operation, and submit the record to the technical manager.

(1) Samples for method blank test

These are samples that shall be prepared for a series of measuring operations. These samples shall be prepared if a major change has been made to the pretreatment operation or at the time of measurement of a high-concentration sample from which inter-sample contamination is expected.

(2) Samples for travel blank test

These samples are prepared by performing a pretreatment operation on the samples subjected to operations for the travel test as prescribed in 4 of Chapter 2.

(3) Samples for dual measurement

These samples are prepared by performing a pretreatment operation on the samples collected for dual measurement as prescribed in 4 of Chapter 2.

(4) Samples of known concentrations

These are reference samples of known concentrations and used periodically to make sure that there is no quality control problem.

Chapter 4 Measurement by gas chromatograph/mass spectrometer (GC-MS)

1. Plan for measuring samples by GC-MS

The measurement technician shall draw up the draft of a plan for measuring samples by GC-MS, and submit it to the technical manager.

The technical manager shall make modifications to the submitted draft as required, and describe it in the draft of the plan for quality assurance and quality control as described in 1 of Chapter 3 in Part 1 as a plan for measuring samples by GC-MS.

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2. Inspection of GC-MS

The technical manager shall make an implementation standard for inspection of GC-MS, submit it to the general supervisor after obtaining the approval of the quality controller, and obtain the approval of the general supervisor. The measurement technician shall perform inspections according to this standard, make a record of the following, and submit the record to the technical manager. The periodical inspection described in (2) refers to inspection or adjustment beyond the category of daily inspection, and can be contracted out. He shall also record measures taken if there are problems such as power failures or malfunctions.

(1) Daily inspection

Person who performed inspection and date of inspection

Basic matters regarding various kinds of wear including the state of use of capillary columns

Basic matters regarding MS including coolant, vacuum pump and degree of vacuum

(2) Periodical inspection

Person who performed inspection and date of inspection

- Circumstances under which inspection was performed
- (3) Maintenance
 - Inspection and replacement of ion source
 - Inspection and adjustment of lens system
 - Adjustment of source slit
 - Inspection and cleaning of interface line
 - Baking (ion source, etc.)
 - Replacement of filament
 - Others
 - (4) Measures taken to deal with problems
 - Details of problems and measures taken

3. Adjustment of GC-MS

The measurement technician shall adjust GC-MS prior to performing the operation as described in 4 or 5, make a record of the following after making sure that GC-MS is ready for use, and submit the record to the technical manager.

- (1) Adjustment of GC
 - Person who made adjustments and date
 - Installation of capillary columns and inspection of carrier gas
- (2) Adjustment of MS
 - Person who made adjustments and date
 - Mass calibration
 - Resolution
- (3) Adjustment of GC-MS
 - Peak separation and absolute sensitivity of GC-MS

4. Creation of calibration curve

In order to make a calibration curve, the measurement technician shall perform a selected ion monitoring operation (SIM operation) on the standard solution for producing a calibration curve, and obtain necessary data. He shall check to see that the obtained data conform with the requirements specified in the measuring procedure, and make a record of the following.

- Person who created calibration curve
- Date of creation of calibration curve
- Intensity ratio for peak area
- Relative sensitivity and its coefficient of variation

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5. Measurement of samples

The measurement technician shall perform a SIM operation on the standard solution for producing a calibration curve, the test samples and the samples prepared in 4 of Chapter 3 according to the plan for measuring samples by GC-MS in order to verify the calibration curve and the variation of sensitivity, thereby obtaining necessary data, and shall make a record of the following. He shall perform the operation for verifying the calibration curve at the time of commencement of measurement each day, and perform the operation for checking the variation of sensitivity at a frequency of more than once a day.

- Person who performed measuring operation
- Date of measurement
- Measuring conditions
- Type and amount of syringe spike added
- Order of measurement (injection list)
- Amount of sample injected

6. Checking calibration curve and checking variation of sensitivity

The measurement technician shall make a record of the following based upon the data obtained from the operation of measuring the standard solution for producing a calibration curve

performed to check the calibration curve and from the operation of measuring the standard solution for producing a calibration curve performed to check the variation of sensitivity as described in 5. In cases where the results do not conform with the requirements specified in the measuring procedure, he shall perform remeasurement after removing the cause(s) (in cases where a problem arises while checking the variation of sensitivity, he shall perform measurement of a series of samples again that he has measured just before the activity), and make a record of the fact that he has made remeasurement and its results.

Results of comparison between the relative sensitivity obtained from the checking operation and the relative sensitivity obtained during the creation of the calibration curve

Variation during retention time

Variation of relative retention ratio to internal standard substance

7. Identification and quantitative determination

The measurement technician shall perform operations for identification and quantitative determination on the data obtained from the SIM operation described in 5 performed on the test samples and the samples prepared as in 4 of Chapter 3, and make a record of the following.

(1) Person who performed operations and date

(2) Check on syringe spike internal standard substance (omit steps described in and after (3) if the check reveals nonconformity with the requirements specified in the measuring procedure)

Comparison between the peak area of syringe spike internal standard substance in the sample and the peak area of syringe spike internal standard substance in the standard solution

Comparison between the result obtained in and the value specified in the measuring procedure

(3) Detection of peaks

Result of check on peak S/N ratio

Peak height

Peak area

(4) Identification of dioxins

Lock mass channel variation status

Comparison between the peak area and the peak area ratio for the standard substance as well as the ion intensity ratio estimated from the isotope abundance ratio

Comparison between the peak retention and the retention time of the standard substance, and comparison between the relative retention time of the corresponding internal standard substance and that of the standard substance

(5) Quantitative determination and calculation of concentration

Concentration calculated

Material explaining the calculation process from which the concentration was obtained

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If there is a need for remeasurement, he shall indicate the reason, make remeasurement, and attach the chart.

Chapter 5 Finalization of results of quantitative determination

The measurement technician shall perform the operation of calculating the lower limit of detection and the lower limit of determination described in 1, and submit the record he made and the records described in 4-7 of Chapter 4 to the technical manager after putting them in order. The technical manager shall examine the submitted records, and when he recognizes no problem with the quality of concentration calculated in (5) of 7 of Chapter 4, he shall inform the measurement technician of the result. If he recognizes any problem, he shall take proper measures under the provision of 2 of Chapter 1 in Part 1.

After being informed by the technical manager that he has recognized no problem with the quality of concentration calculated as in (5) of 7 of Chapter 4, the measurement technician shall perform the operations as described in 2-6 below, and submit the records he made to the technical manager.

performed to check the calibration curve and from the operation of measuring the standard solution for producing a calibration curve performed to check the variation of sensitivity as described in 5. In cases where the results do not conform with the requirements specified in the measuring procedure, he shall perform remeasurement after removing the cause(s) (in cases where a problem arises while checking the variation of sensitivity, he shall perform measurement of a series of samples again that he has measured just before the activity), and make a record of the fact that he has made remeasurement and its results.

Results of comparison between the relative sensitivity obtained from the checking operation and the relative sensitivity obtained during the creation of the calibration curve

Variation during retention time

Variation of relative retention ratio to internal standard substance

7. Identification and quantitative determination

The measurement technician shall perform operations for identification and quantitative determination on the data obtained from the SIM operation described in 5 performed on the test samples and the samples prepared as in 4 of Chapter 3, and make a record of the following.

(1) Person who performed operations and date

(2) Check on syringe spike internal standard substance (omit steps described in and after (3) if the check reveals nonconformity with the requirements specified in the measuring procedure)

Comparison between the peak area of syringe spike internal standard substance in the sample and the peak area of syringe spike internal standard substance in the standard solution

Comparison between the result obtained in and the value specified in the measuring procedure

(3) Detection of peaks

Result of check on peak S/N ratio

Peak height

Peak area

(4) Identification of dioxins

Lock mass channel variation status

Comparison between the peak area and the peak area ratio for the standard substance as well as the ion intensity ratio estimated from the isotope abundance ratio

Comparison between the peak retention and the retention time of the standard substance, and comparison between the relative retention time of the corresponding internal standard substance and that of the standard substance

(5) Quantitative determination and calculation of concentration

Concentration calculated

Material explaining the calculation process from which the concentration was obtained

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If there is a need for remeasurement, he shall indicate the reason, make remeasurement, and attach the chart.

Chapter 5 Finalization of results of quantitative determination by GC-MS

The measurement technician shall perform the operation of calculating the lower limit of detection and the lower limit of determination described in 1, and submit the record he made and the records described in 4-7 of Chapter 4 to the technical manager after putting them in order. The technical manager shall examine the submitted records, and when he recognizes no problem with the quality of concentration calculated in (5) of 7 of Chapter 4, he shall inform the measurement technician of the result. If he recognizes any problem, he shall take proper measures under the provision of 2 of Chapter 1 in Part 1.

After being informed by the technical manager that he has recognized no problem with the quality of concentration calculated as in (5) of 7 of Chapter 4, the measurement technician shall perform the operations as described in 2-6 below, and submit the records he made to the technical manager.

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Chapter 6 Report of results

After being informed by the technical manager that he has finalized the concentration according to the procedure described in Chapter 5, the measurement technician shall perform the operations listed below, and submit records he made to the technical manager after putting them in order. The technical manager shall check the submitted records, retain them, prepare the draft of a report on the results of quality assurance and quality control as described in 2 of Chapter 3 in Part 1, and submit the draft to the quality controller.

1. Indication of measurement results

He shall indicate the measurement results obtained from the calculation of concentration, data of which have been finalized as in Chapter 5, by means of the method specified in the measuring procedure, and record them. He shall use an indication method so that concentrations of measured metamers that are above the lower limit of detection but below the lower limit of determination can be easily identified.

2. Calculation of toxic equivalents

He shall obtain toxic equivalents by means of the calculation method specified in the measuring procedure, and record the obtained values. In addition, he shall prepare material, such as coefficients of toxic equivalents he used, that explains the calculation process from which he obtained the toxic equivalents.

3. Causes of abnormal and missing values

For abnormal and missing values that made it impossible to finalize the data in Chapter 5, he shall investigate their causes, and record the results of his investigation.

4. Storage of samples

He shall retain and manage samples for remeasurement in accordance with the plan for quality assurance and quality control, and record their control numbers, storage and management methods and periods.

Attached Table 1 Measurement items and methods for measuring environmental dioxins as covered in these Guidelines

| Item | Measurement method |
|---|---|
| Constant monitoring or survey of ambient air (Monitoring of compliance with air quality standards, etc.) | Table attached to Environment Agency Notification No. 68 of 1999 (Method for measuring, by means of a high-resolution gas chromatograph mass spectrometer, samples collected by means of an air sampler equipped with a polyurethane-foam fitted sampling canister at the rear of the filter) Regarding the enforcement of the Law concerning Special Measures for Dioxins (Notifications) 2(2)b(a) of III in Kankiki No. 11, Kanhoan No. 6, Kandaiki No. 11, Kandaiki No. 5, kansuiki No. 14, kansuikan No. 1, Kansuiki No. 5, Kansuido No. 7 issued in 2000 (hereinafter referred to as "Enforcement Notification") |
| Constant monitoring or survey of water quality (water quality of public water bodies and underground water) (Monitoring of compliance with water quality standards, etc.) | Table attached to Environment Agency Notification No. 68 of 1999 (Method specified in K0312 of the Japanese Industrial Standard) Enforcement Notification 2(2)b(b) of III |
| Constant monitoring or survey of the status of soil pollution (Monitoring of compliance with soil quality standards, etc.) | Table attached to Environment Agency Notification No. 68 of 1999 (Method for Soxhret extraction of dioxins contained in soils and measurement by a high-resolution gas chromatograph mass spectrometer) Enforcement Notification (2(2)b(c) of III) |
| Survey of the status of pollution by emissions | Prime Minister's Office Ordinance No. 67 of 1999 (Method specified in K0311 of the Japanese Industrial Standard) |
| Survey of the status of pollution by effluents | Prime Minister's Office Ordinance No. 67 of 1999 (Method specified in K0312 of the Japanese Industrial Standard) |
| Survey of the status of pollution by dust, incineration ash and other combustion residue | Ministry of Environment Notification No. 80 of 2004 (Method specified by the Minister of Environment pursuant to the provisions of Article 1, Paragraph 2 and Item 1 of the Enforcement Ordinance of the Law Concerning Special Measures for Dioxins) |
| Survey of the status of pollution by sediment of public water body (Monitoring of compliance with sediment quality standards, etc.) | Manual for the Survey and Measurement of Dioxins in Sediment (March 2000, Environment Agency) (Method for Soxhret extraction of dioxins contained in sediments and measurement by a high-resolution gas chromatograph mass spectrometer) |

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| 1. Basic documents | |
| Documents on organization | Part 1, Chapter 1, 1(1) |
| Structural chart of organization | Ditto |
| Procedures for dealing with QC problems | Part 1, Chapter 1, 2 |
| Procedure for preparing, maintaining and controlling documents/records | Part 1, Chapter 1, 5 |
| Standard operating procedures | Part 1, Chapter 2, 1 |
| Document on operating conditions in facilities | Part 2, Chapter 1, 5 |
| Standard for implementing inspections of GC-MS | Part 2, Chapter 4, 2 |
| 2. Plans, reports, etc. | |
| Internal audit report | Part 1, Chapter 1, 3 |
| Quality improvement instructions | Ditto |
| Document for implementing quality improvements | Ditto |
| Report on training | Part 1, Chapter 1, 4 |
| Plan for quality assurance and quality control | Part 1, Chapter 3, 1 |
| Report on the results of quality assurance and quality control | Part 1, Chapter 3, 2 |
| 3. Records | |
| Record of task progress status | Part 1, Chapter 2, 2 |
| Record of reagents | Part 2, Chapter 1, 1 |
| Record of standard substance (solution) | Part 2, Chapter 1, 2 |
| Record of instruments | Part 2, Chapter 1, 3 |
| Record of apparatuses | Part 2, Chapter 1, 4 |
| Results of preliminary survey on sample collection | Part 2, Chapter 2, 1 (1) |
| Record of the process leading to decision not to perform sample collection | Part 2, Chapter 2, 2 |
| Record of sample collection | Part 2, Chapter 2, 3 |
| Record of the conditions under which sample collection was performed for travel blank test and dual measurement | Part 2, Chapter 2, 4 |
| Record of acceptance inspection of samples | Part 2, Chapter 3, 2 (1) |
| Record of storage and management of samples | Part 2, Chapter 3, 2 (2) |
| Record of extraction from samples, etc. | Part 2, Chapter 3, 2 (3) |
| Record of cleanup of extract from samples, etc. | Part 2, Chapter 3, 2 (4) |
| Record of preparation of samples to be measured along with test samples | Part 2, Chapter 3, 4 |
| Record of inspection of GC-MS | Part 2, Chapter 4, 2 |
| Record of adjustment of GC-MS | Part 2, Chapter 4, 3 |
| Record of creation of calibration curve | Part 2, Chapter 4, 4 |
| Record of measurement of samples | Part 2, Chapter 4, 5 |
| Record of checks on calibration curve and variation of sensitivity | Part 2, Chapter 4, 6 |
| Record of identification and quantitative determination | Part 2, Chapter 4, 7 |
| Record of lower limit of detection and lower limit of determination | Part 2, Chapter 5, 1 |
| Record of recovery | Part 2, Chapter 5, 2 |
| Record of field blank test | Part 2, Chapter 5, 3 |
| Record of travel blank test | Part 2, Chapter 5, 4 |
| Record of dual measurement | Part 2, Chapter 5, 5 |
| Record of measurement of samples of known concentration | Part 2, Chapter 5, 6 |
| Record of indication of measurement results | Part 2, Chapter 6, 1 |

| | |
|---|----------------------|
| Record of calculation of toxic equivalents | Part 2, Chapter 6, 2 |
| Record of causes of abnormal and missing values | Part 2, Chapter 6, 3 |
| Record of storage of samples | Part 2, Chapter 6, 4 |

Attachment 1 Plan for quality assurance and quality control

The plan for quality assurance and quality control described in Part 1, Chapter 3, 1 shall contain the following matters.

I. General matters

1. Title and control number of the plan
2. Table of contents
3. Description of the nature of the plan
4. Name and address of organization that performs the tasks
5. Summary of dioxin measurement tasks to be performed
6. Official title, name and signature of the general supervisor, and the date on which he signed his name
7. Scheduled implementation period for each process of the tasks
8. Official titles and names of quality controller, technical manager and measurement technician
9. Name and address of the client
10. Tasks allotted to other organizations (when applicable)

II. Sample collection plan (Part 2, Chapter 2, 1)

1. Person who collects samples
2. Scheduled sample collection date
3. Sampling sites
4. Need for preliminary survey (if needed, summary of the preliminary survey)
5. Sampling instruments and apparatuses and reagents to be used
6. Summary of sample collection operation
7. Sample containers
8. Means of transportation after collecting samples
9. Plan for performing travel blank test and dual measurement

III. Sample pretreatment plan (Part 2, Chapter 3, 1)

1. Acceptance inspection (inspector, scheduled inspection date and details)
2. Storage and management of samples until extracting operation (place, method, period)
3. Extracting operation (operator, scheduled extraction date, method and conditions)
4. Type and amount of cleanup spike to be added, and time to add cleanup spike
5. Cleanup of extract from samples (operator, scheduled cleanup date, method and conditions)
6. Preparation of samples to be measured along with test samples

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IV. Plan for measuring samples by GC-MS (Part 2, Chapter 4, 1)

1. Adjustment of GC-MS
2. Creation of calibration curve
3. Measurement of samples
4. Work related to checking of calibration curve and variation of sensitivity
5. Work related to identification and quantitative determination

V. Finalization of results of quantitative determination of GC-MS (Part 2, Chapter 5)

1. Work related to calculation of lower limit of detection and lower limit of determination
2. Work related to checking of quality of concentrations calculated
3. Work related to calculation of recovery and checking of results

4. Work related to field blank test, travel blank test, dual measurement and calculation/verification of results of measurement of samples of known concentrations
5. Work related to finalization of results of quantitative determination on test samples

VI. Report of results (Part 2, Chapter 6)

1. Indication of measurements
2. Handling of abnormal and missing values
3. Storage of samples, etc.

The report on the results of quality assurance and quality control described in Part 1, Chapter 3, 2 shall contain the following matters.

I. General matters

1. Title and control number of the report
2. Table of contents
3. Description of the nature of the report
4. Name and address of organization that performed the tasks
5. Summary of dioxin measurement tasks performed
6. Official title, name and signature of the general supervisor, and the date on which he signed his name
7. Implementation period for each process of the tasks
8. Official titles and names of quality controller, technical manager and measurement technician
9. Name and address of the client
10. Tasks allotted to other organizations (when applicable)
11. Indication of each page to ensure that the report has no missing pages
12. Indication about the final page

II. Sample collection

1. Record of preliminary survey (Part 2, Chapter 2, 1(1))
2. Record of sample collection (Part 2, Chapter 2, 3)
3. Conditions under which samples were collected for travel blank test and dual measurement (Part 2, Chapter 2, 4)

III. Sample pretreatment

1. Acceptance inspection (Part 2, Chapter 3, 2(1))
2. Storage and management of samples until extracting operation (Part 2, Chapter 3, 2(2))
3. Extracting operation (Part 2, Chapter 3, 2(3))
4. Cleanup of extract from samples (Part 2, Chapter 3, 2(4))
5. Preparation of samples to be measured along with test samples (Part 2, Chapter 3, 4)

IV. Measurement of samples by GC-MS

1. Daily inspection, periodical inspection and maintenance of GC-MS (Part 2, Chapter 4, 2)
2. Adjustment of GC-MS (Part 2, Chapter 4, 3)
3. Creation of calibration curve (Part 2, Chapter 4, 4)
4. Measurement of samples (Part 2, Chapter 4, 5)
5. Checks on calibration curve and variation of sensitivity (Part 2, Chapter 4, 6)
6. Identification and quantitative determination (Part 2, Chapter 4, 7)

V. Finalization of results of quantitative determination

1. Lower limit of detection and lower limit of determination (Part 2, Chapter 5, 1)
 - (1) Process of calculating lower limit of detection and lower limit of determination for apparatus, and basic calculation data
 - (2) Process of calculating lower limit of detection and lower limit of determination for measuring method, and basic calculation data
 - (3) Process of calculating lower limit of detection and lower limit of determination at the time of sample measurement, and basic calculation data

(4) List of lower limits of detection and lower limits of determination

| | Calculated value | Required value |
|--|------------------|----------------|
| Lower limit of detection for apparatus | | |
| Lower limit of determination for apparatus | | |
| Lower limit of detection for measuring method | | |
| Lower limit of determination for measuring method | | |
| Lower limit of detection at the time of sample measurement | | |
| Lower limit of determination at the time of sample measurement | | |

2. Recovery (Part 2, Chapter 5, 2)

(1) Cleanup spike recovery

The following table shall be completed, and if there is any problem with recovery, the related factual data shall be recorded.

[Timing of adding clean up spike:]

| Cleanup spike Name of internal standard substance | Cleanup spike Amount added | Cleanup spike Recovery | Remarks |
|--|-------------------------------|---------------------------|---------|
| | | | |
| | | | |
| | | | |
| | | | |

(2) Sampling spike recovery

The following table shall be completed, and if there is any problem with recovery, the related factual data shall be recorded.

| Sampling spike Internal standard substance | Sampling spike Amount added | Sampling spike Recovery | Remarks |
|---|--------------------------------|----------------------------|---------|
| | | | |
| | | | |
| | | | |
| | | | |

3. Blank tests (Part 2, Chapter 5, 3 and 4)

(1) Conditions under which the field test blank was performed, results of the test and evaluation of the results

(2) Conditions under which the travel blank test was performed, results of the test and evaluation of the results

4. Conditions under which dual measurement was conducted, results of the dual measurement and evaluation of the results (Part 2, Chapter 5, 5)

5. Verification through measurement of samples of known concentrations (Part 2, Chapter 5, 6)

(1) Source of samples of known concentration, etc.

(2) Comparison of measurement results between measurement performed this time and measurements performed in the past

6. Conditions under which the results of quantitative determination on test samples were finalized (if results that could not be finalized were obtained, the facts to that effect shall be described, and the details shall be provided in VI, 3.)

VI. Report of results

1. Measurement results (Part 2, Chapter 6, 1)

2. Toxic equivalents (Part 2, Chapter 6, 2)

(1) Coefficient of toxic equivalents used

(2) Calculated results of toxic equivalents

3. Abnormal and missing values (Causes of the results that could not be finalized in V. 6 shall be described) (Part 2, Chapter 6, 3)

4. Storage of samples (Part 2, Chapter 6, 4)

VII. Attached documents

1. Structural chart of organization

2. All chromatograms

3. Material for confirming variation of lock mass channel

4. Material for confirming resolution

5. Injection list

The following matters shall be recorded with regard to Part 2, Chapter 2, 3 (2).

1. Sampling instruments and apparatuses, and reagents used
 - Manufacturer, model name and simulated illustration of high-volume air sampler used
 - Conditions under which instruments and parts etc. are cleaned, and conditions of their storage until use
 - Suction pump (model name, name of manufacturer)
 - Flowmeter (Type, model name, name of manufacturer and calibration results)
 - Type of filter paper and absorbent (names of product and manufacturer, lot number or properties), cleaning method and results of check on blank
2. Sampling operation
 - High-volume air sampler installation site and its conditions (height from ground, etc.)
 - Summary of sampling operation
 - Type and amount of sampling spike, timing to add sampling spike, period until sample collection and storage conditions
 - Time when sampling started and ended, and flow rate
3. Sample containers
 - Container for storing filter paper after sampling and frequency, etc. of cleaning of the container
4. Other additional matters
 - Weather, temperature, atmospheric pressure, humidity and wind direction/velocity on the sampling day and the previous day
 - Obstacles around the sampling site

The following matters shall be recorded with regard to Part 2, Section 2, 3 (2).

1. Sampling instruments and apparatuses, and reagents used

Manufacturer, model name and simulated illustration of water sampler and water sampling equipment

Frequency, etc. of cleaning of water sampler, and the conditions of its storage before use

Material of water sampling equipment and its conditions until use (whether it was in constant use or not in use, etc.)

2. Sampling operation

Summary of sampling operation

Site where samples were collected (depth of water, etc.)

Structure and depth of wells, position of strainers, etc. (in the case of underground water)

When the pump is used to sample water: time required from the start-up of the pump to the start of water-sampling, and pump discharge (in the case of underground water)

3. Sample containers

Material, volume, cleaning status and conditions of their storage until use

4. Other additional matters

Weather at the time of sampling and on the previous day (in cases where it rained hard a few days earlier, also record the situation)

Temperature and water temperature at the time of sampling

Conditions of samples (color, odor and other basic matters that indicate the conditions of sampled water)

In the case of rivers, their flow rate

The following matters shall be recorded with regard to Part 2, Chapter 2, 3 (2) and Chapter 3, 3.

1. Sampling

(1) Selection of sampling sites

Existence and location of buildings and trees around sampling sites, surrounding conditions including sunshine

Ground conditions at sampling sites

Sampling method and distance between sampling sites

(2) Sampling instruments and apparatuses, reagents used

(3) Sampling operation

Summary of sampling operation (depth)

Characteristics of soil samples (color, properties, dirt, etc.)

(4) Sample containers

Material, volume, cleaning status and conditions of their storage until use

(5) Other additional matters

Weather on the day before the sampling day

2. Sample pretreatment

(1) Air drying of samples

Method, place and time

(2) Sifting

(3) Completion of equivalent mixture

(4) Water content and ignition loss

The following matters shall be recorded with regard to Part 2, Chapter 2, 3 (2) and Chapter 3, 3.

1. Sampling

(1) Sampling instruments and apparatuses, reagents used

Manufacturer, model name and simulated illustration

Sampling tube (material, nozzle inner diameter, and use or non-use of a cooling device)

Filter material

Liquid collector (number of absorption bulbs, volume, type and amount liquid absorbed)

Suction collector (shape of absorbent column, and material, product name and quantity of absorbent)

Suction pump (model and manufacturer names)

Flowmeter (type, model and manufacturer names, and calibration results)

(2) Sampling operation

Preliminary survey (height of sampling site from ground, conditions of hole for measurement, location of blower/exhauster, and shape of duct)

Set amount of sample gas to be collected, sampling time and uniform suction flow rate

Conditions under which leak test was conducted and its results

Temperature and pressure of gas meter

Temperature at filter collector and liquid collector

Uniform suction flow rate, suction time and amount of gas suctioned

Type, amount of sampling spike, time to add sampling spike, period and storage conditions until sampling

Amount of gas sampled

Temperature, flow rate, composition, pressure, amount of water content, etc. of emissions

(3) Sample containers

Sample recovering method

Sample storage method (material, volume, etc. of sample containers)

(4) Other additional matters

Type of source of samples collected and its operating conditions

Correction based on oxygen concentration (applicable to waste incinerators, etc.)

2. Sample pretreatment

(1) Sulfur acid treatment of collected dust

The following shall be recorded with regard to Part 2, Chapter 2, 3 (2).

1. Sampling instruments and apparatuses, reagents used
 - Manufacturer and model names, and simulated illustration of water sampler
 - Frequency, etc. of cleaning of water sampler, and its storage conditions until use
2. Sampling operation
 - Summary of sampling operation
3. Sample containers
 - Material, volume and frequency, etc. of cleaning of sample containers, and their storage conditions until use
 - Sample storage method (material, volume, etc. of sample containers)
4. Other additional matters
 - Weather on the day before the sampling day
 - Temperature and water temperature at the time of sampling
 - Sample conditions (color, odor and other general matters regarding water quality)
 - Type of source of samples collected, and conditions of its use
 - Effluent passage route, including the name of the river into which it was discharged

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Attachment 8 Individual matters related to dust and incineration ash, and other combustion residues

The following matters shall be recorded with regard to Part 2, Chapter 2, 3 (2) and Chapter 3, 3.

1. Record of sampling

(1) Sampling instruments and apparatuses, reagents used

Type and material of sampling instruments

(2) Sampling operation

Sampling sites

Summary of sampling operation

(3) Sample containers

Type and material of sample containers

(4) Other additional matters

Sample conditions

Operating status of source of samples collected (incineration plant)

2. Sample pretreatment

(1) Sample preparation method (pulverization information, particle size, etc.)