

Chapter 7

Environmental Quality Analysis

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Abstract Environmental quality assessments require the determination of concentrations of various substances. This chapter outlines key points for environmental quality analysis. First, environmental standard values (environmental standards and regulation standards) and analytical methods (official methods) are explained. Next, preparation methods for analytical samples are discussed with respect to sampling methods and sample preparation. Samples must be suitable for analysis after preparation. Pretreatment of samples for analysis is discussed in Sect. 6. Analytical values are obtained by the analysis of pretreated samples, and the reliability of the analytical values is described in Sect. 7. Finally, a leaching test, used for waste analysis, is discussed in terms of judgment criteria for landfill waste, environmental quality standards for soil pollution, soil leaching and content standards, and radioactive substance standards.

Keywords Environmental standards • Official method • Sampling • Pretreatment • Reliability of analytical values • Leaching test

7.1 Introduction

Environmental quality analysis is conducted for various reasons, and thus types of environmental samples vary greatly, including water, air, soil, and waste samples. Environmental quality analysis also covers a wide range of processes, including the preparation of a sampling plan and its execution, transportation and storing of samples, chemical analysis (pretreatment and measurement), and statistical treatment of data and interpretation. Among these processes, chemical analysis requires a complicated analytical method for each sample, due to the fact that there are diverse chemical substances with different concentrations and properties in samples, and some substances may interfere with analysis of other substances.

The first half of this chapter on environmental quality analysis introduces environmental standards, official methods, data reliability, and pretreatment of samples, and the second half of this chapter discusses a case involving waste

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analysis. There are various kinds of waste analysis, including hazard judgment tests of wastes, content tests for recycling, and trace analysis. This chapter describes a leaching test, a type of hazard judgment test.

7.2 Environmental Standards

With the aim of protecting human health and conserving the environment, environmental standards (ESs) are used to establish benchmarks for air, water, soil, and noise quality. Benchmarks are then used to create policies that will maintain those ESs. ESs serve as a “desired goal,” i.e., a target used by governments to establish environmental policies. They are not representative of the minimum level needed to maintain human health and other conditions; rather, they are a set of targets that are meant to be actively maintained. For areas in which pollution is not currently taking place, ESs should be established in order to maintain the status quo and therefore avoid any decline from the current state ([Website of the Ministry of the Environment](#)). ESs are defined in Japan’s Basic Environment Law (Basic Environment Law Section 3 “Environmental Quality Standard”). In the Basic Environment Law, concrete values of standards are established for air (Environmental Quality Standards for Air), noise (Environmental Quality Standards for Noise, Environmental Quality Standards for Aircraft Noise, and Environmental Quality Standards for Shinkansen Super-express Railway Noise), water (Environmental Quality Standards for Water and Environmental Quality Standards for Groundwater), and soil (Environmental Quality Standards for Soil) quality. Concrete values of standards are also defined for dioxins released through air pollution, water contamination (including contamination of the seafloor), and soil pollution under the Act on Special Measures against Dioxins ([Website of the Ministry of the Environment](#)).

7.3 Regulation Standards

Regulation standards (RSs) are a set of standards to regulate behavior that causes pollution as defined by laws or ordinances. Facilities such as factories are required to comply with these standards. Terms vary depending on the laws, i.e., the RSs are called “emission standards” in the Air Pollution Control Act, “effluent standards” in the Water Quality Pollution Control Act, and “regulatory standards” in the Noise Regulation Law, the Vibration Regulation Law, and the Offensive Odor Control Law. RSs are generally imposed to maintain ESs.

7.3.1 Stringent Standards

National governments establish RSs for the emission of dust, soot, and toxic substances into the air and various effluents into the water. Prefectural governments are allowed to establish more stringent standards than national standards through ordinances based on their judgment of natural and social conditions. Such standards are called stringent standards (SSs).

7.3.2 Total Amount Regulation

Total amount regulation (TAR) is a method used to assign the allowable emission of pollutants and contaminants from factories, thereby limiting the total level of pollutants and contaminants in a certain area to an allowable level in terms of environmental conservation. The allowable level is determined by multiplying the gas emission volume by the concentration of the pollutant for air pollution and by multiplying the drainage volume by the concentration of the contaminant for water contamination. TAR is enforced in specific areas for sulfur oxides and nitrogen oxides in the air and for chemical oxygen demand (COD), nitrogen, and phosphorus in the water.

7.4 Official Method

An “official method” is important for environmental quality analysis and frequently used by governments for performing environmental monitoring. For example, standard values and measurement methods for dioxins are defined by ESs related to air pollution, water contamination (including contamination of the seafloor), and soil pollution by dioxins ([Website of the Ministry of the Environment](#)). Similarly, water quality is based upon Japanese Industrial Standards (JIS) K 0312 (2008). A “measurement manual” is also established for an “official method,” defining detailed measurement methods. The use of an “official method” is necessary for analytical values to become official values, and a certified environmental measurer must issue a measurement certification. The measurement certification also verifies the ability of the measurer.

7.5 Sample Preparation

7.5.1 Sampling (Collecting Samples)

When sampling is performed for analysis, it is important that the sample is collected without bias and is representative of the whole population of interest. Generally, the whole population of environmental samples is heterogeneous, and therefore the sampling technique requires careful attention. Increasing the number of samples will improve the estimation of the representative value of the whole population, but the cost of sampling and time constraints may make it difficult to collect more samples.

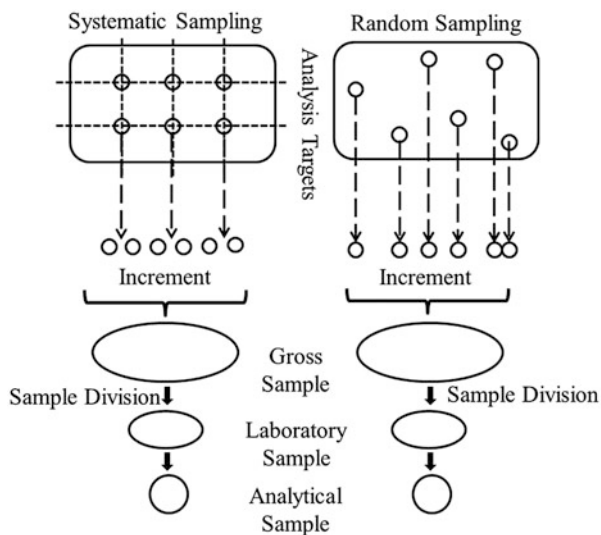
Types of sampling methods include random sampling and systematic sampling. In the former method, samples are chosen randomly and entirely by chance; in the latter method, samples are collected at a constant interval of time and space. There are also other sampling methods, such as two-stage sampling, and an appropriate sampling method should be chosen based upon the state of the samples.

7.5.2 Sample Preparation

A lot, or batch, is a set of materials subjected to analysis that come from the same source, with nearly uniform properties and constituents. The amount of sample collected from the lot by a sampler in a single action is called an increment. For example, a solid sample (e.g., soil) is tested using a shovel designed for increment sampling, and each collected increment is pulverized separately. All increments are combined into a bulk sample, or gross sample, in order to obtain a representative sample with average composition for the whole analytical population and then subjected to “sample division” using either an increment sample division method, a method with a riffle sampler, or a sample division apparatus to obtain laboratory samples. Sample division is a process by which a single sample is divided into smaller groups of samples with the same physical properties or chemical constituents. Divided samples are then used as analytical samples. Figure 7.1 illustrates the sampling and sample preparation procedure for soil. The details for sampling and sample procedure for wastes are shown in JIS K 0060 (1992).

Once the samples from the bulk sample are used up, it is very difficult to obtain identical environmental samples with the same properties at a later time. The common practice for analysis is, therefore, to refrain from using up the samples during the preparation procedure and set aside a portion of the samples.

Fig. 7.1 Soil sampling and sample preparation procedures



7.6 Pretreatment

Increments collected from analytical populations by sampling are transformed into analytical samples through a series of techniques. Raw samples are subjected to analysis if they can be used directly for instrumental analysis or titration. However, samples usually require pretreatments such as decomposition, dissolution, extraction and concentration of the target constituent, and the removal of inhibiting constituents. Main techniques of pretreatment are detailed below.

7.6.1 Decomposition and Dissolution

If an analytical sample is converted into a solution, the sample is processed by wet decomposition, generally using an acid (e.g., hydrochloric acid, nitric acid, sulfuric acid). Stronger acids often used in wet decompositions include perchloric acid and hydrofluoric acid. Perchloric acid must not be used alone on organic compounds because the reaction is explosive and very dangerous. Hydrofluoric acid is also hazardous. If hydrofluoric acid adheres to and then penetrates the skin, it causes a chemical burn that is difficult to heal. Therefore, gloves are mandatory when using hydrofluoric acid. Inhaling the vapor of hydrofluoric acid is even more hazardous. When decomposing a sample, the reagent must not interfere with the following analytical techniques. In the past, heating during decomposition was conducted using an electric furnace or a hot plate, but have been replaced by microwave extraction in recent years. In this method, samples are sealed in a Teflon pressure decomposition container and decomposed by irradiating microwaves. Therefore,

this method prevents the volatilization of acids and can decompose the sample with a small amount of acid in a short period of time. Another decomposition process is the dry ash method. However, this process is an open system that carries the risk of pollution and scattering of samples and acid. Therefore, a closed system has been favored for pretreatment in recent years.

7.6.2 Extraction from Solids

The pretreatment method in which an analytical target substance (analyte) is taken out of a solid sample is called extraction. A simple method involves combining the sample with a solvent, pulverizing with a homogenizer to uniformity, mixing with a shaker, and then filtering. Soil samples are typically treated using a Soxhlet extractor. When using a Soxhlet extractor, a sample is placed on a cylindrical filter paper and extracted with an organic solvent through a cycle of repeated evaporation and condensation. Other methods of extraction from solids include supercritical fluid extraction.

7.6.3 Liquid-Liquid Extraction

Transferring analytes or interfering substances in a sample solution to another solvent is called liquid-liquid extraction. Solvents immiscible with each other are added into a separable funnel with the sample, and the separable funnel is then shaken until the two layers form an interface and reach the distribution equilibrium. The solution is then left standing, until the interface separates completely, and the layer of interest is taken out. Generally, organic compounds are transferred from an aqueous solution to an organic solvent. In the case of metal analysis, chelating agents may be used to extract the metal as the metal chelates.

7.6.4 Solid Phase Extraction

Solid phase extraction is a separation method in which a gas or liquid sample is passed through a solid phase to trap chemical constituents. The use of solid phase extraction is expanding for use in other applications, such as the removal of coexisting substances by use of different adsorbents (solid phase) or solvents, in addition to the separation (extraction) of analytes. Solid phase extraction methods include the batch method, column method, and membrane method, although the cartridge method is most commonly used. Various solid phases have been used in studies, which include a normal-phase type, an ion-exchange type, a chelate type, and a reverse-phase type. The cartridge is first conditioned by washing with the

solvent to wet the surface, and then the sample solution is added to the cartridge. After the sample has been loaded into the cartridge, impurities are removed through washing with a nonpolar solvent. Finally, the analytes are eluted with a polar solvent.

7.6.5 Concentration

Concentration is a process by which some amount of solvent is removed from a solution to increase the concentration of the target solute in the solution, used in cases where only trace amounts of the analyte exist in the solution sample. Concentration is necessary especially when the concentration is lower than the minimum detectable concentration (limit of detection) of the analytical instrument. A common practice is to use a rotary evaporator, in which the sample solution is heated, rotated, and depressurized, and the solvent is evaporated and recovered by cooling.

For a small amount of solution sample, nitrogen gas is blown in to evaporate the solvent to concentrate the solution. A Kuderna-Danish (KD) concentrator and a centrifugal evaporator are also used. However, these concentration methods have a drawback, as they also concentrate coexisting substances along with the analytes. It is possible to separate coexisting substances by a liquid-liquid extraction method or solid phase extraction method, in addition to concentration.

7.6.6 Other Pretreatment Methods

Distillation Distillation is a fundamental pretreatment method that has been used since the early days of chemical measurements. Types of distillation include steam distillation and reductive distillation. Distillation can remove impurities that may interfere with analysis. Distillation separates component substances from liquid mixtures through vaporization and condensation based on different volatilities, and thus, highly pure analytes can be obtained.

Headspace Method and Purge-and-Trap Method The headspace method and purge-and-trap pretreatment methods utilize the process of gasification. In these methods, the gas phase is taken out and analyzed using gas chromatography or other instruments. The headspace method gasifies the analytes when the sample liquid is sealed in a container. The sample rests until it reaches a constant concentration based on its partition coefficient. The sample is then taken out by a gas phase syringe. This method is widely practiced due to its ease of use.

Gel Permeation Chromatography (GPC) Gel permeation chromatography (GPC) is a type of size exclusion chromatography in which molecules are sifted

according to their size. The method is able to separate analytes from sample-derived fat constituents.

Other methods of pretreatment include filtration, dialysis, precipitation, and recrystallization.

7.7 Reliability of Analytical Values

7.7.1 Uncertainty

Conventionally, an error has been defined as the “difference between the measured value and true value”; however, a “true value” is a conceptual value that cannot actually be obtained. “Uncertainty” is a term proposed by several international organizations, including the International Organization for Standardization (ISO), as a unified expression of the reliability of measurement results, with the idea that a range of measured quantity is obtained from the dispersion of the data using known measurement results.

With the proposal of the concept of uncertainty, the definition of “reliability” in JIS K 0211 was also updated as the expression of the concrete range of the measured quantity, from “the degree of precision or correctness expected” (1987 version) to “tempestable property or degree to maintain specified functions and performance of instruments, methods, and their elements within the boundary of a given set of conditions” (2013) (JIS K 0211 [2013](#)).

7.7.2 Technical Terms Related to Reliability

The degree of dispersion of measured values is called “precision,” while the degree of deviance from the true value is called “trueness.” The concept that combines precision and trueness is called “accuracy.” The quantitative expression of accuracy is “uncertainty.” While the degree of deviance of measured values from the true value was referred to as “correctness” in the past, it is referred to as “trueness” today in order to avoid confusion between “correctness” and “accuracy.” Precision is further divided into repeatability and reproducibility. Repeatability is the degree of consistency in values, when the same analyst continuously repeats measurements using the same method on the same day. Reproducibility is the degree of consistency in values, when the date and time, analyst, and/or methods are different. Reproducibility yields greater dispersion than repeatability. However, different technical terms may be used in place of these technical terms in some fields, and verification is necessary in each field (this section uses the terminology from the field of chemical measurements).

7.8 Leaching Test Method

Analyses of wastes are roughly classified into leaching tests and content tests. Analyses related to landfill disposal are subjected to leaching tests, which are at the center of waste analysis. A leaching test is the tool used to assess the environmental impact of wastes when the wastes are buried in landfill or recycled. The leaching test method varies by country; this section introduces the official method in Japan.

7.8.1 *Judgment Criteria for Landfill*

Announcement No. 13 by the Ministry of the Environment (“Test Method for Metals in Industrial Wastes,” partially revised in 2013) outlines the method of judging the hazard of toxic substances leached from wastes buried in a landfill. The leaching test defined in the announcement is as follows.

A waste sample is prepared by sifting to obtain particles with diameters of 0.5–5 mm. Pure water (A3 or A4 in JIS K 0557) (JIS K 0557 1998) is used as the solvent. The sample and pure water are mixed at a liquid-solid ratio (L/S, the ratio of 10 g of solid to 1 mL of liquid) of 10. The mixture is subjected to horizontal shaking for 6 h. After shaking, the resulting mixture is subjected to centrifugal separation at a 3000 gravitational acceleration for 20 min. Filtration is performed with a 1 μm membrane filter, and the filtrate is finally submitted for analysis.

L/S is set at 10 with the idea that a criterion is set for the hazard, when waste materials come in contact with ten times the weight of water to the waste (Sakai et al. 1996).

7.8.2 *Environmental Quality Standards for Soil Pollution*

A leaching test method for measuring environmental quality standards for soil pollution is defined in Announcement No. 46 by the Ministry of the Environment (“On the Environmental Quality Standards for Soil Pollution”). This method is also sometimes used as a judgment method to assess suitability when waste-derived materials are recycled for secondary use for purposes such as construction materials. Samples with particle diameter of 2 mm or smaller are prepared by sieving and then subjected to horizontal shaking under the same conditions as those in Announcement No. 13 by the Ministry of the Environment. The sample is centrifugally separated, and the filtrate is obtained by filtration with a 0.45 μm membrane to be submitted for analysis.

7.8.3 *Soil Leaching and Content Standards*

A measurement method for soil leaching is defined in “On Determining a Measurement Method for Soil Leaching Surveys (2003 Ministry of the Environment Announcement No. 18).” The quantity of leaching is measured by the appended measurement method from Announcement No. 18 by the Ministry of the Environment, using testing liquid prepared by the method in Announcement No. 46 by the Ministry of the Environment. The appendix of Announcement No. 46 defines measurement methods by the types of specific toxic substances. This measurement method assumes a scenario in which toxic substances in soil pollute the groundwater.

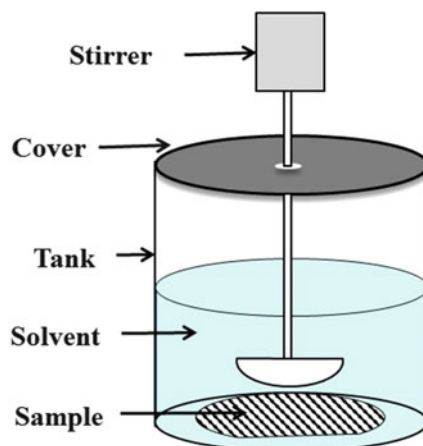
A measurement method for soil content is defined in “On Determining a Measurement Method for Soil Content Surveys (2003 Ministry of the Environment Announcement No. 19).” At least 6 g of the sample as in Announcement No. 46 (sample with 2 mm or less particle diameter prepared with sieve) is used, and the sample is mixed with a solvent, 1 mol/L of hydrochloric acid (unit mL), at 3 % of the weight-volume ratio. The mixture is shaken at 200 rpm for 2 h by a shaker and allowed to stand for 10–30 min. The filtrate is obtained by filtration of the supernatant from the mixture with a membrane filter of 0.45 μm pore diameter to be submitted for analysis. The amount of heavy metals leached into the hydrochloric acid is calculated as the amount per 1 kg of dry sample weight. This measurement method is used to assess the risk for oral intake of metals in the soil.

7.8.4 *Radioactive Substance Standards*

A great amount of wastes containing radioactive substances were generated due to environmental pollution of radioactive substances released from the Fukushima power plant disaster, caused by the Great East Japan Earthquake on March 11, 2011. For these wastes containing radioactive substances, the leaching test method in JIS K 0058-1 (2005) is used, rather than the methods in Announcement No. 13 by the Ministry of the Environment. This is the method selected to test the leaching property of radiation at waste treatment sites under conditions similar to those in the field. The procedure is detailed in the “Waste Material Guidelines Part 5” of “A Guideline for Measuring Radioactive Concentration” (2011).

Figure 7.2 shows the schematic drawing of the leaching test apparatus. In this method, solvent, ten times the weight of water to the sample, is added to a certain amount of sample, and the resulting mixture is stirred at 200 rpm for 6 h to leach chemical substances (e.g., cesium) and obtain the testing liquid. After stirring for 6 h, the sample is allowed to stand for 10–30 min, and the resulting dispersion is removed from the tank. Removed liquid is centrifugally separated at 3000 rpm for 20 min as needed, and the filtrate of the supernatant is obtained by filtration with a membrane filter of 0.45 μm to be submitted for analysis.

Fig. 7.2 Schematic drawing of the leaching test apparatus



7.9 Conclusion

This chapter provides an overview of some of the key points in environmental analyses. However, types of environmental samples are diverse and include water, air, soil, and waste samples, and analytical methods are equally diverse. This chapter does not go into extensive details, due to space constraints. The reader is referred to the references for further details (Hirai 2006; Ichikuni 1998; Tsunoda 2013; Tsumura 2009). It is very important to consider the quality and quantity of samples for assessing the environment. This chapter is to be used as a reference, and in conjunction with detailed measurement manuals developed by the Japanese Industrial Standards Committee and the Ministry of the Environment, highly reliable analyses may be conducted for environmental impact assessments.

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