Determination of Substances of Concern Regulated in EU

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In recent years, advanced regulations concerning restrictions on the use of hazardous substances have been enforced in the EU, as environmental pollution control acts. Subsequent to the ELV for automobiles, restrictions on the use of hazardous substances are being examined for the field of electric and electronic equipment, where both WEEE and RoHS directives have been enacted. In the ELV, the use of four heavy metals (Cadmium, Lead, Mercury, Chromium) has been restricted. The use of the same heavy metals as in the ELV, as well as two additional flame retardants (Polybrominated biphenyl, Polybrominated diphenyl ether) has been restricted in RoHS. Under these circumstances, the control of certain hazardous substances in products takes on great importance.

This paper describes the use of various analytical equipment in screening analysis by XRF and precision analysis for Cd, Pb, Hg, Cr, Br.

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Introduction

The European Union (EU) is currently comprised of 25 countries. The population of the EU is approximately 450 million, far exceeding that of the U.S.A.'s estimated 300 million. Its GDP has also reached approximately US$1 trillion, which is nearly equivalent to that of the U.S.A., thus indicating its considerable economic power. Accordingly, many policies stipulated by the EU have had a dramatic effect on communities and practices throughout the world.

The EU clarified its basic environmental policies in Article 174 (Objectives of EU Environmental Policy) of the Treaty on European Union. The following topics of policy are included in the article: preservation, protection and improvement of environmental quality; protection of human health; placing top priority on eliminating causes of environmental disruption; and the "polluter pays" principle. Sophisticated, future-oriented measures for control of environmental pollution are being taken, based on these fundamental policies.

Stimulated by the EU's movement, many countries are now devising regulations to control the use of specific substances of concern. **Table 1** indicates the regulations to restrict the use of substances of concern in EU member countries and other nations around the world.

Table 1 Restrictions on substances of concern

country/area	
EU	Waste electrical and electronic equipment (WEEE) Restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS) End of life vehicles (ELV)
Japan	The marking for presence of the specific chemical substances for electrical and electronic equipment (J-Moss)
USA (California)	Electronic Waste Recycling Act (S.B.20) Electronic Waste, Advaced Disposal Fees (S.B.50)
USA (CONEG)	The Model Toxics in Packaging Legislation[1]
Korea	The Act for Resource Recycling of Electrical/Electronic Products and Automobiles (Korean WEEE, RoHS,)
China	Management Method on the Prevention and Control of Pollution Caused by Electronic inforamation Products (Chinese RoHS)

Table 2 Maximum concentration values of substances of concern

unit : mg/kg

	Substance	ELV	RoHS	Procurement guideline	Procurement guideline
Heavy metal	Pb	1000	1000	100	250
	Hg	1000	1000	—	1000
	Cd	100	100	5	50
	Cr^{6+}	1000	1000	—	1000
Flame retardant	PBB		1000	—	1000
	PBDE		1000	—	1000

Table 2 indicates the types of substances of concern and their maximum concentration values.

Under the ELV, a maximum concentration value has been stipulated for each of four heavy metals: 1,000 ppm for lead (Pb), mercury (Hg), hexavalent chromium (Cr^{6+}) and 100 ppm for cadmium (Cd). Under the RoHS, a value of 1,000 ppm has been specified as the maximum concentration for two brominated flame retardants (i.e. polybrominated biphenyl and polybrominated diphenyl ether) in addition to the above four heavy metals stipulated under the ELV.

The impetus for Japan to pay serious attention to regulations, such as WEEE and RoHS, was an incident that occurred in October 2001. The incident was that Cd in an amount exceeding the value stipulated by the chemical control law by the Netherlands (in effect since June 1996) was detected in electronic game machines manufactured in Japan. As a result the game machines could not pass the customs process and in turn distribution of those products was made impossible. Japanese manufacturers of electric/electronics equipment suffered considerably due to this incident and were pressed to take measures in order to comply with regulations of the countries with which they were dealing. Some manufacturers of game machines established even more stringent standards for parts and materials than those stipulated by the EU, causing enormous confusion in the relevant industries. In addition to these game machine makers, certain electric/electronics equipment manufacturers began setting their own standards.

In response to the EU regulations, parts and material suppliers for electric/electronics equipment manufacturers and the automobile industry are finding it necessary to demonstrate that their products contain either no substances of concern or those having amounts less than the regulated values. These efforts are referred to as "green procurement," "green purchasing" or "green partnering," and have become increasingly prevalent in Japan. Under these circumstances it has also become critically important to analyze for the presence of regulated hazardous substances in products.

Analytical System for Substances of Concern

Fig. 1 depicts an example of the analytical system for the detection and quantitative analysis of substances of concern.

An analytical system is usually comprised of two stages: the first stage for screening and the second stage for specific quantitative analysis. In the first stage the screening analysis is conducted using energy-dispersive x-ray florescence spectrophotometer (EDXRF) to determine the concentration of substances of concern in the sample. Although such concentrations can be detected quickly and readily using EDXRF, there are significant discrepancies in the values indicated. Therefore, when a regulated element exceeding the detection limit has been detected, it is better to conduct an individual quantitative analysis in order to obtain a more accurate result. Currently, however, determining the concentration using the threshold value alone is being considered as well. **Table 3** shows the equation for the threshold values when judging the determined concentrations using the threshold values. In such calculations the threshold value can be determined with consideration for the safety coefficient (%)

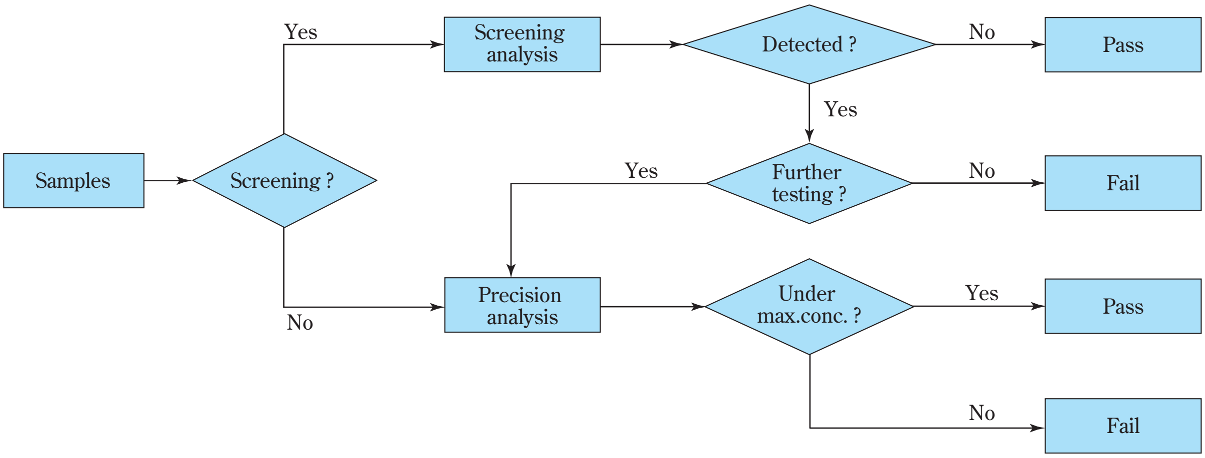


Fig. 1 Analyical system

and reproducibility (σ). If the detected value is lower than the lower limit, the sample is deemed to have passed the test; if it is higher than the upper limit, it is deemed to have failed. If the result is somewhere between "passed" and "failed" (i.e. the gray zone), individual quantitative analysis will be conducted as part of the second stage, for a more accurate result. However, this procedure will not be applied to Cr or Br, since these chemicals are regulated depending on the valences and chemical species.

Table 3 Screening limits for regulated elements

Element	Lower limit	Upper limit
Cd, Pb, Hg	C × (1 – S) – nσ	C × (1 + S) + nσ
Br, Cr	C × (1 – S) – nσ	—

C : Maximum concentration value
S : Safety coefficient
σ : Reproducibility
n : Constant

In the second stage of analysis an inductively coupled plasma atomic emission spectrometer (ICP-AES) or inductively coupled plasma mass spectrometer (ICP-MS) or absorption spectrophotometer is used for the metal components, such as Cd, Pb, Hg and Cr^{6+}. For Br and bromine compounds an ion chromatography (IC) or gas chromatograph mass spectrometer (GC-MS) is used to obtain highly accurate values for the regulated hazardous substances.

In some cases it is obvious that a more precise analysis of the second stage is required, right from the beginning.

Specific analytic procedures are described in the following section.

1. Screening Analysis

First, an EDXRF screening analysis is conducted prior to the precision analysis. Although several days are required to obtain the results of the precision analysis, with EDXRF screening the presence of the regulated hazardous substances can be confirmed on the same day. In order to save time and costs, it is important to select samples via the screening analysis, instead of conducting a precision analysis on every sample.

The most popular EDXRF that is currently used in laboratories is a desktop type equipped with a 50W x-ray tube and an Si semiconductor detector. Therefore, its detection accuracy for impurities is superior to that of a portable EDXRF. Additionally, since it has a quantitative lower detection limit ranging from 10 to 50 ppm; it demonstrates an accuracy sufficient for detection of the regulated values stipulated by the ELV and similar regulations. However, its performance isn't sufficient to detect the threshold values of Cd that have been established by each respective company. Precautions must be taken in such cases, since it's necessary to conduct a precision analysis during the initial stage.

The desktop EDXRF can detect the regulated con-

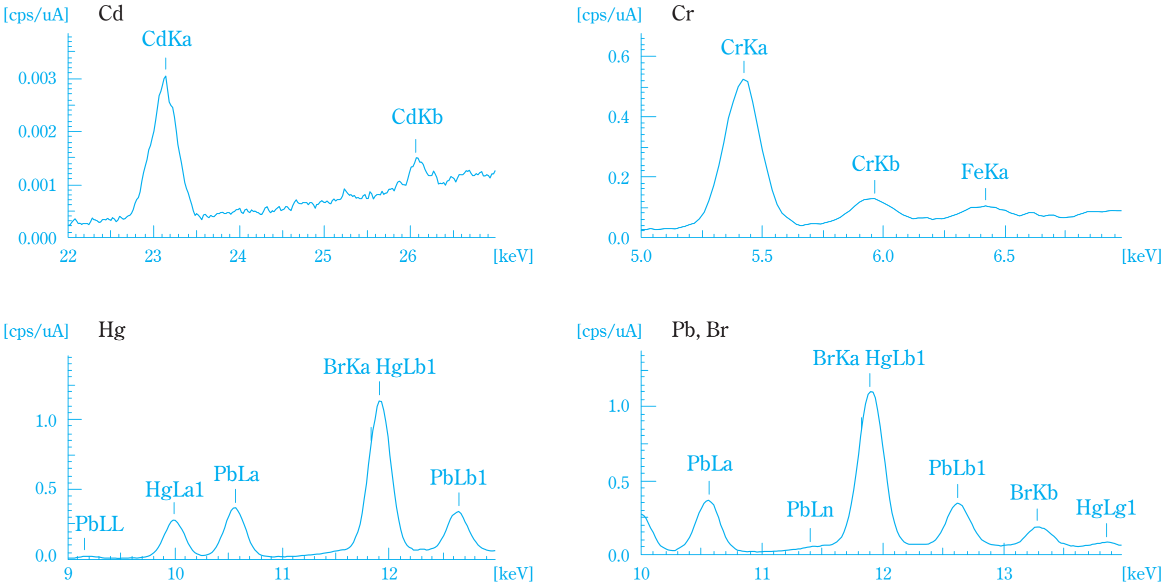


Fig. 2 Analytical results of EDXRF

centrations of Cd, Pb, Hg, Cr and Br in the atmosphere. In fact, not only can it detect these element concentrations in solid samples, it can also detect these element concentrations in powder samples and large samples, depending on the structure of the procedure used. Furthermore, if a measuring cup is used for a liquid sample, it can be used for measurements in exactly the same way as it is used with solid samples. Thus, the versatility of the desktop type EDXRF has been enhanced.

Although the detection time varies according to the conditions of analysis, only from a few minutes to just over 10 minutes is required. Results can be obtained in the form shown in **Fig. 2**. The presence of the subject element can be detected through the presence of a peak in the energy position of the subject element.

When using the EDXRF, a correlation is observed between the peak intensity and the concentration of the element. It is therefore possible to detect the concentration based on the peak intensity. **Fig. 3** indicates the calibration lines for a PVC resin sample containing the subject elements. The results show that EDXRF can detect subject element concentrations up to 10 to 50 ppm, although this varies according to the particular element.

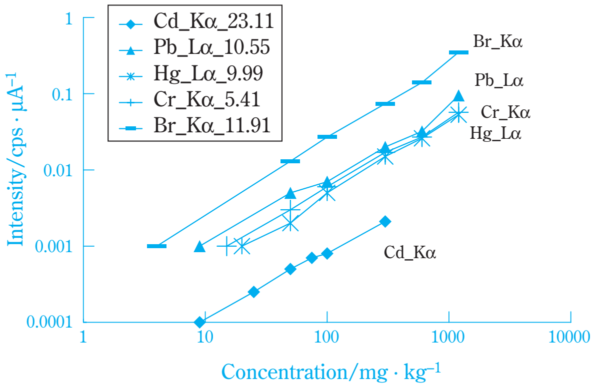


Fig. 3 Calibration of elements in standard sample of PVC

The intensity of EDXRF can be significantly affected by the excitation and absorption effects of the elements of the sample, such as the matrix. Therefore, when a matrix element is different for samples, even though several subject elements exist in the exactly the same concentrations, their EDXRF intensities aren't always the same. **Fig. 4** depicts the calibration lines for Cr obtained from the tests using PVC and LDPE resin samples. The inclinations of the calibration lines differ considerably, detecting that the intensity will also differ greatly, depending upon the type of resin (such as PE and PVC), even though the same concentration of Cr has been added to each sample. Therefore, when detecting the concentration of subject elements using EDXRF, attention must be paid to the matrix of the sample and the calibration lines must be selected carefully, so as to secure results. Moreover, because EDXRF intensity can be significantly affected by the sample characteristics (such as thickness, surface condition and particle shape, etc.), a precision analysis, as described in the previous section, is required in order to ensure an accurate determination of any samples within the gray zone.

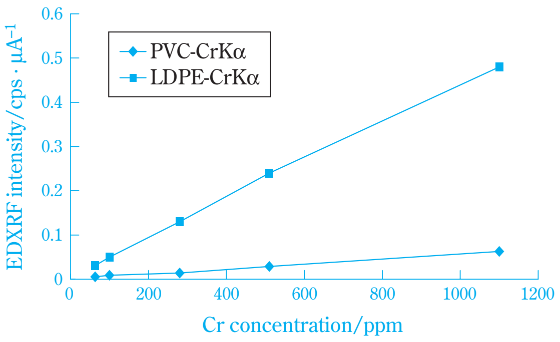


Fig. 4 Comparison of X-ray strength in different matrix

Additionally, because EDXRF cannot identify trivalent and hexavalent chromium, analysis values are obtained as total Cr (T-Cr). Thus, in the event that Cr is detected exceeding the value determined as a gray zone, a specific analysis for Cr^{6+} must be conducted. Moreover, if Br is detected in concentrations exceeding the value determined as a gray zone, then analysis for PBB and PBDE must be conducted using a GC-MS or similar apparatus.

2. Precision Analysis of Regulated Hazardous Substances

(1) Precision Analysis for Cd, Pb, Hg, Cr and Br

The general pretreatment method, when assaying metals contained within organic compounds, is wet acid digestion using mixed reagents of nitric acids/sulfuric acids.[2)] However, the analysis samples involved with ELV and RoHS (i.e., parts for vehicles, electric/electronics equipment) are composed not only of plastics but also of various other components, such as metals and ceramics. Therefore, when using the aforementioned mixed reagents, mere decomposition of the sample is often inadequate.

Infusible residues can adversely affect the accuracy of such analysis and occasionally even a coprecipitation of the substances of concern can occur. It is therefore crucial to prevent the loss of any regulated hazardous substances and to ensure that they are dissolved completely. Moreover, a variety of decomposition methods should be prepared for the testing of special samples.

In this paper we will introduce a precision chemical analysis method for Cd, Pb, Hg, Cr^{6+}, T-Cr and Br. Furthermore, the precision chemical analysis for T-Cr and Br may occasionally be conducted prior to the precision analysis of Cr^{6+}, PBB and PBDE, when omitting a screening analysis.

(i) Cd, Pb, T-Cr

The dissolution of the sample involves wet acid digestion using mixed reagents of nitric acid, hydrochloric acid, hydrofluoric acid and hydrogen peroxide. This dissolution process can occasionally be accelerated by microwave radiation (heat decomposition) and mountable decomposition under pressure. The ashing method (decomposition through combustion) is occasionally used as a means for analyzing organic samples. For the analysis of infusible residues and samples that do not decompose easily, fusing agent or strong oxidizing agents, such as perchloric acid, can be employed.

Because the sample solutions obtained through the above procedures contain acids, fusing agent and main components of the sample, a considerable load is placed on the analytical apparatus, possibly altering the measurement sensitivity of such apparatus, or causing interference. If interference is recognized, measurements must be conducted using one of the following methods: the matrix matching method, using a standard solution to which the main components of the sample have been added in order to obtain calibration lines; the internal standard method; and the standard addition method. For the measurement of metals contained within the sample solution, ICP-AES, ICP-MS or ET-AAS is used.

Fig. 5 depicts the flow diagram of the analytical procedures that combine the microwave sample digestion method and the fusion/acid dissolution method. This is an effective method of analysis used when infusible residues are produced following the microwave sample digestion process. Several procedures must be conducted, depending on the sample. **Table 4** depicts the analytical results produced from an actual sample. From the results, it is quite obvious that if decomposition of the infusible residue was not conducted, the concentration of the subject element could be underestimated.

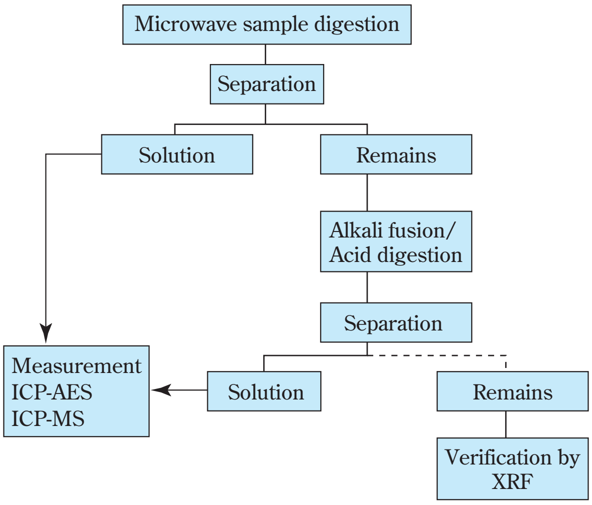


Fig. 5 Flow diagram of pretreatment and analysis

Table 4 Analytical results of Cr in polymer

unit : mg/kg

	Cr (n = 1)	Cr (n = 2)
Microwave sample digestion (The sample was dissolved incompletely)	1,200	660
Microwave sample digestion and alkali fusion/acid digestion (The sample was dissolved completely)	5,500	5,500

(ii) Hg

As previously described, because the wet acid digestion method uses a strong oxidizing agent, such as nitric acid or perchloric acid, it produces a large amount of hydrogen ions as the decomposition of the organic compound progresses. Given such conditions, although the temperature is low, Hg has sufficient volatility that it may vaporize and be dispersed outside of the system during this procedure. For that reason it is necessary to use a mountable system or employ a reflux condenser. To determine Hg within a sample solution, ICP-AES, ICP-MS or CV-AAS is used.

(iii) Cr^{6+}

The toxicity of Cr differs according to the valence, being either trivalent or hexavalent. Because hexavalent Cr has higher toxicity than trivalent Cr, and it is more readily absorbed into the human body,[3)] specific evaluation is required. However, when dissolving a sample using wet acid digestion, Cr becomes oxidized. Therefore, one should not conduct any pretreatment using an oxidizer.

Given the difficulty of dissolving a sample while at the same time maintaining a hexavalent status, Cr^{6+} can be extracted for analysis using water, taking advantage of the fact that it is easily dissolved in water.

When extracting Cr^{6+} it is necessary to use an appropriate extract, in accordance with the particular sample. For metal samples, hot water is used as an extract. For non-metal samples, alkali solutions are often used.

Cr^{6+} within the extract reacts with diphenylcarbazide as a color indicator, and forms a purplish-red chromium diphenylcarbazone complex. An absorption spectrophotometer is used to determine Cr^{6+} concentration in the solution.

Given certain types of samples, colored components can transfer to the extract. In some cases, even though a color indicator has been added, coloration may not occur due to the presence of organic matter. If coloration fails to occur, it is mandatory to confirm the coloration of the extract and recovery rate after the coloration, which is caused by the addition of an appointed amount of standard sample.

(iv) Br

Br can be collected using either of the following methods: the combustion collection method, employing a combustion tube in which the sample is subjected to combustion decomposition in the oxygen flow, whereupon the combustion gas is collected through the bubbling method using a weak alkaline solution; or the oxygen cylinder method, in which the sample is subjected to combustion decomposition in a sealed container filled with oxygen, whereupon the combustion gas is absorbed into a weak alkaline solution. IC is used to determine the Br concentration in the solution.

The latter method makes it possible to determine the Br derived from PBB and PBDE, as well as the Br derived from other bromine compounds. Therefore, when a screening analysis is not performed, this method can be performed prior to a precision chemical analysis for PBB and PBDE.

(2) Analysis Examples

Table 5 indicates the analytical results of a standard sample of polyethylene by a variety of pretreatment methods. The analytical values match the certified values of reference to a significant extent.

Table 5 Analytical results of Cd,Cr,Hg,Pb and Br in polyethylene (BCR-680)

unit : mg/kg

Element	Certified value	ICP-MS	ICP-AES	CV-AAS	ET-AAS	IC
Cd	141	145	143	—	—	—
Cr	115	122	110	—	—	—
Hg	25	26	—	25	—	—
Pb	108	109	—	—	113	—
Br	808	—	—	—	—	792

Analysis : Pretreatment

ICP-MS : Mountable acid digestion
ICP-AES : Demountable acid digestion
CV-AAS : Acid digestion attached reflux condenser
ET-AAS : Mountable acid digestion
IC : Combustion in the atmosphere of oxygen

3. Analysis of Brominated Flame Retardants

Many parts and components of items that are indispensable to a comfortable life, such as vehicles, home electrical appliances and OA equipment, are made of organic polymers, such as rubber and plastics. This is because organic polymers offer excellent workability and insulation. They also help to save weight and cost. Additionally, the thermal resistance and flame resistance of such materials has been improved and for that reason their use has been greatly expanded.

The flame retardants used for organic polymers can be categorized as inorganic, bromide, chloride or phosphate. In Japan, the most popular flame retardant among these is of the inorganic type, followed by bromide and phosphate types. The least popular retardant is the chloride type.[4)]

Table 6 indicates the major flame retardants and their associated compounds.

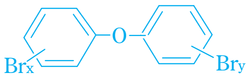
Table 6 The classification of flame retardant

Type of compound	Compound
Inorganic	Antimony (Ⅲ) Oxide, Magnesium hydroxide, Zinc borate
Bromide	Deca brominated biphenyl ether, Tetrabromobis phenol-A, Hexabromo benzene
Chloride	Chlorinated paraffine, Chlorinated polyethylene
Phosphate	Triphenyl phosphate, Tricresyl phosphate
Others	Triazine compounds, Guanidine compounds, Silicone oil

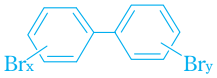
Needless to say, each flame retardant has its own unique flame-resistant effects. However, it is known that synergistic effects can be obtained by combining several flame retardants. The combination most often used to achieve better flame resistance is that of a halogenated flame retardant and antimony trioxide. However, other combinations, such as phosphorus compounds and nitrogen compounds, as well as metal hydroxides and metal nitrate salts, are often employed.5) These flame retardants have become indispensable as a means of improving the flame resistance of organic polymers.

However, although many chlorinated or brominated flame retardants are very toxic in the human body, they are found in a wide variety of common materials. Thus, they are attracting tremendous attention worldwide, as chemical substances that may proliferate on a global scale and cause serious environmental contamination. Moreover, because these flame retardants produce dioxins when combusted, a worldwide movement is gaining momentum to regulate the use of these retardants. PBB and PBDE have been designated as specified brominated flame retardants under the RoHS and it is required that their content be below regulated values.

Table 7 indicates the number of isomer of the chemical formulae of PBB and PBDE shown in **Fig. 6**. PBB and PBDE are chemical substances in which ten hydrogen atoms in two phenyl groups have been replaced with one to ten bromines. There are 209 isomers in PBB and PBDE. The PBB and PBDE compounds most commonly used as flame retardants are those based on Deca compounds.



Polybrominated diphenyl ether (PBDE)
$x + y = 1\sim10$



Polybrominated biphenyl (PBB)
$x + y = 1\sim10$

Fig. 6 The chemical formula of PBDE and PBB

Table 7 Name of homologue and number of isomer of PBBs and PBDEs

Number of bromine	Name of homologue	Number of isomer
1	Mono brominated	3
2	Di brominated	12
3	Tri brominated	24
4	Tetra brominated	36
5	Penta brominated	42
6	Hexa brominated	36
7	Hepta brominated	24
8	Octa brominated	12
9	Nona brominated	3
10	Deca brominated	1
Total		209

The types of samples subject to RoHS analysis are quite diverse: raw materials, molded products and composite materials, both organic and inorganic. However, because their characteristics and interfering ingredients differ depending upon the sample, one cannot apply the same pretreatment method in every case. In this paper we will introduce the pretreatment method that is most often stipulated under RoHS regulations, with respect to organic polymers.

(1) Pretreatment Procedures for Organic Polymers

Specified brominated flame retardants are usually mixed into organic polymers. Therefore, when performing a pretreatment on an organic polymer, it is important to extract a specified brominated flame retardant from the material. Of further importance is both the refining process used to remove any interfering ingredients that have been extracted together with the specified brominated flame retardant, and the concentrating process used to detect even a minute amount of flame retardant. The pretreatment procedures introduced below have been commonly undertaken for the determination of additives contained within organic polymers.

(i) Extraction

The organic polymer (referred to as “polymer” hereafter) is first dissolved in a solvent, in order to allow the specified brominated flame retardant (referred to as “flame retardant” hereafter) to separate from the polymer. This solution is then slowly dripped into the solvent, such as methanol, in which the polymer does not easily dissolve, so as to allow the polymer to be deposited. By separating this deposited polymer from the solvent through filtration, the flame retardant can be isolated and extracted into the solvent. In this method, selecting the appropriate solvent and controlling the conditions (such as the amount of solvent and the drip rate) are the most critical factors in achieving the best recovery of the flame retardant.

If the polymer doesn’t dissolve in the solvent, it is soaked in a solvent in which the flame retardant can be readily dissolved (such as acetone or n-hexane), and then the solvent is subjected to ultrasonic vibration, or is circulated using a Soxhlet extractor, to ensure that the flame retardant dissolves in the solvent. It is important, when performing these procedures, to extract an adequate amount of flame retardant by combining several solvents, thereby increasing the permeability of the polymer into the solvent, or by making the polymer into a finely pulverized powder, thereby obtaining a larger surface area where it can come into contact with the extracting solvent.

(ii) Refining

Refining is performed to remove any interfering ingredients from the extraction solution. This process is very important, given that it influences both the accuracy of identification and the determined sensitivity. The refining method most commonly used is the treatment by concentrated sulfuric acid. In this method the interfering ingredients are decomposed by the concentrated sulfuric acid, and the decomposition product is dissolved in concentrated sulfuric acid then removed by separating solvent. Because the interfering ingredients and extracting solvent might react strongly with the concentrated sulfuric acid, safety is extremely important when performing this treatment. Moreover, a column chromatography treatment, which uses activated silica gel to absorb and separate the interfering ingredients, can occasionally be employed. In this method, it is necessary to understand the behavior of the flame retardant, in order to optimize the conditions for elution. It is also important to obtain repeatability in the elution behavior of the flame retardant, by dehydrating the extract prior to the column chromatography treatment.

(iii) Concentration

The concentration is commonly carried out by the evaporator method, in which the extracting solvent is volatilized under a vacuum. In this method, a small amount of toluene, in which the flame retardant dissolves more readily than the extracting solvent, is added to inhibit vaporization.

(2) Identification and Determination

One of the conditions of identifying flame retardants is that the retention time matches that of a standard substance. However, for PBBs and PBDEs, standard substances are not always available for all isomers. Therefore, identification of the peak is performed because the retention times are adjoining the obtained homologues.

Another condition of identification is that the natural isotope ratio of bromine matches that of a standard substance. Specifically, M^+, $(M+2)^+$ and $(M+4)^+$ for each homologue are first detected using the selected ion monitoring method and if the peak area ratio of $(M+2)^+$ and $(M+4)^+$ to M^+ is ±20% that of the standard substance, it is deemed to consist of PBBs and PBDEs.

A determination must be performed for each isomer using the calibration curve from the chromatogram areas of M^+, $(M+2)^+$ and $(M+4)^+$. To create a calibration curve, more than one type of isomer is used for each homologue.

Because homologues that have large numbers of bromines have high boiling points, it is necessary to use a special separation column that has a greater heat resistance than is standard.

(3) Decomposition by Light and Heat

Because PBDEs are photolytic, it is important to use a brown-colored container for pretreatment, if possible. If a transparent container is used for pretreatment, it is important to avoid exposure to sunlight. **Fig. 7** depicts the decomposition process when exposed to sunlight for both a DecaBDE compound placed in a transparent container and a DecaBDE compound placed in a brown container. While decomposition began slowly with the start of exposure in a transparent container, with more than 20% of the DecaBDE decomposing after 10 hours, no decomposition was recognized in the brown-colored container.

Fig. 7 also shows the time course of the DecaBDE placed in the transparent container, under exposure to fluorescent light. Decomposition was not observed, which proves that pretreatment can be performed under fluorescent light in a room.

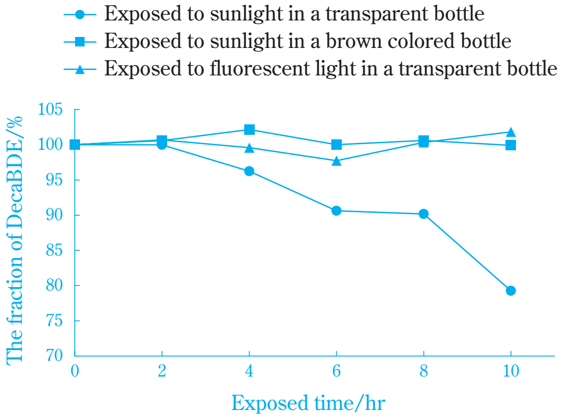


Fig. 7 The decomposition of DecaBDE by exposure to sunlight

Additionally, since PBDEs possess pyrolytic properties, it is important to select an appropriate vaporization temperature for the gas chromatograph. **Fig. 8** depicts changes to the amount of NonaBDEs produced through the thermal decomposition of DecaBDE, which is caused by changes to the sample vaporization temperature of the gas chromatograph. Also, the evaporation behavior of the DecaBDE at each temperature is indicated by its peak area.

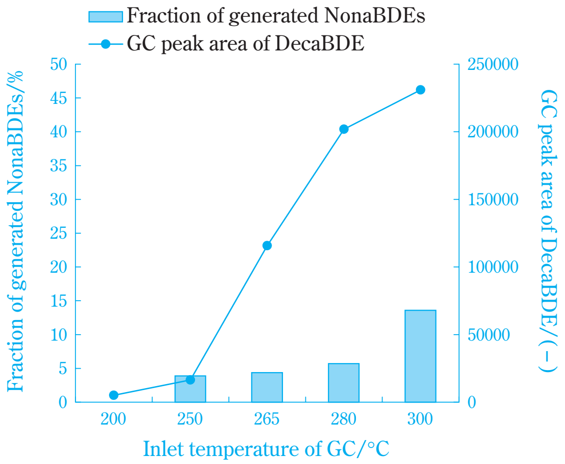


Fig. 8 Thermal decomposition of DecaBDE in GC [6)]

Although the production of NonaBDEs due to thermal decomposition is not recognized at a temperature of 200°C, the amount of decomposition gradually increases at higher temperatures. At a temperature of 300°C, 15% of the DecaBDE has decomposed to NonaBDEs. Contrastingly, although the amount of vaporization of DecaBDE is adequate at 300°C, temperatures below 280°C cause vaporization to decrease substantially, rendering conditions inadequate for microdetermination. From these factors it can be concluded that the optimum sample vaporization temperature is 280°C, in order to minimize thermal decomposition, yet ensure sufficient vaporization of DecaBDEs.

Accordingly, to analyze substances having large numbers of isomers and various properties (such as PBB and PBDE), it is necessary to use technology to optimize the conditions for pretreatment and analysis.

(4) Analysis Example Using an Actual Sample

Fig. 9 depicts a chromatogram from the GC-MS for an example in which a flame retardant has been detected from a wire-coating material. Among these peaks, 107 ingredients have been identified as TriBDEs ~ DecaBDEs. However, their total percentage is merely 0.1%, so this concentration is considered to be too small for the flame retardants to have been added deliberately. Therefore, this wire-coating material was assumed to be made of a recycled material containing flame retardants.

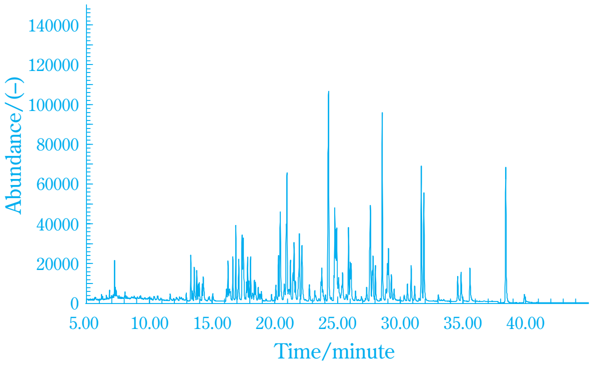


Fig. 9 Analysis of coverig material on electric wire

Conclusion

There are great adverse effects from the hazardous substances contained in products with short service lives and large production volume (such as home electrical/electronic appliances and vehicles). These adverse effects become serious issues that can affect our quality of life whenever these hazardous substances are disposed of, recovered and recycled. This issue will continue to be of great importance for human beings in the future.

Although this paper has discussed only six hazardous substances, the regulations will be increasingly expanded and will also become more stringent. It is therefore mandatory to establish measurement methods that describe the process in detail so as to comply with regulations. It is urgent that such measurement methods be standardized. We will establish a system by which we can respond to these situations precisely, quickly and affordably.

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