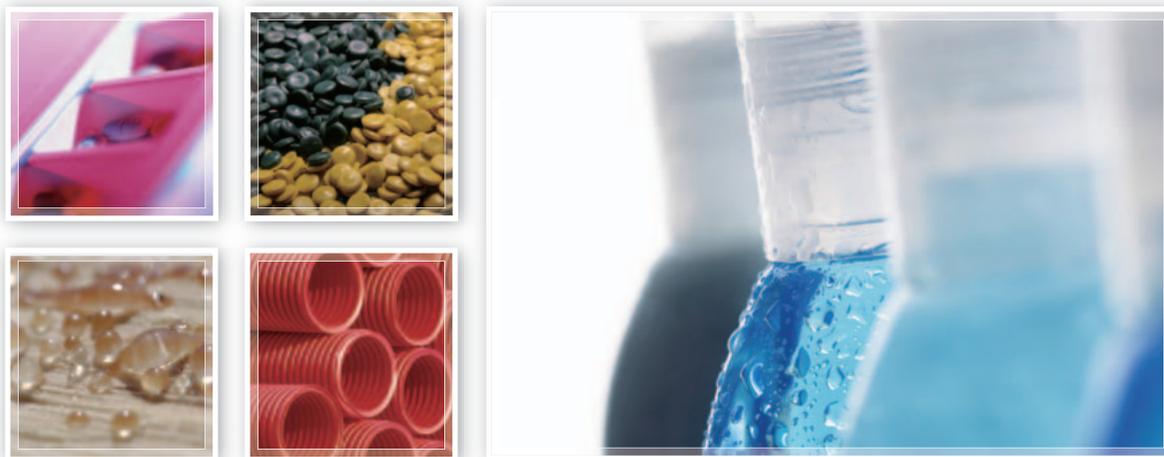


Solutions for Plastic Evaluation



Index

Evaluation of Plastic Materials

Analysis purpose	Application	Instrument	Page
Quality Control	Determination of Hydroxyl Value in Polypropylene Glycol by NIR-PLS Method	FTIR	4
Particle Size Control and Particle Size Distribution	High-Sensitivity, High-Reproducibility Measurement of Single-Digit Nanoparticles Dispersed in Liquid	Single Nano Particle Size Analyzer	5

Evaluation of Raw Materials

Analysis purpose	Application	Instrument	Page
Molecular Weight Distribution	Example of Recycling Preparative Separation of Polystyrene	HPLC	6
Structure and Composition	FTIR Measurement of Plastic Pellets and Powder by Single Reflection ATR Method	FTIR	7
Structure and Isomers	FTIR Analysis of Polybutadiene Microstructure Using Single Reflection ATR	FTIR	8
Structure and Copolymers	Quantitation of Vinyl Acetate in EVA	FTIR	9
Additives	Analysis of Additives in Plastic	GC/MS	10
Additives	Analysis of Additives in Plastic Using LCMS-2020	LCMS	11
Additives	Analysis of Additives in Polymer Extract Using LCMS-IT-TOF (Part 1)	LCMS-IT-TOF	12
Additives	Analysis of Additives in Polymer Extract Using LCMS-IT-TOF (Part 2) – Predicting the Structure of Additives and Attributing MS3 Measurement Results –	LCMS-IT-TOF	13
Additives	Analysis of Additives in Acrylic Sheets	MALDI-TOFMS	14
Additives	Quantitation of Reinforcing Materials in Epoxy Resins	Thermal Analysis	15
Evaluation of Impurities and Byproducts	Analysis of Trace Homopolymer Components in Copolymers	MALDI-TOFMS	16
Impurities and Residues	Analysis of Residual Organic Solvents in Flexible Packaging Materials	GC	17

Product Evaluation

Analysis purpose	Application	Instrument	Page
Surface Structure	Observing the Lamellar Structure of Polymer Film Using a Scanning Probe Microscope	SPM	18
Surface Structure	Analysis of Film Surface by FTIR-ATR Method	FTIR	19
Local Structure	Measurement of Water Melting in a Polymer Electrolyte Fuel Cell Membrane Using a DSC	Thermal Analysis	20
Local Structure	Near-Infrared Mapping of Adhesives Using an Infrared Microscope	Infrared Microscope Systems (FTIR)	21
Optical Properties	Measurement of Diffuse Reflectance in Plastic Using an Integrating Sphere	UV	22
Orientation	Measurement of Polarized Light of PET Film Using FTIR-ATR Method	FTIR	23
Crystallization	Measurement of Commercial PET Bottles	Thermal Analysis	24
Thermal Characteristics	Measurement of Thermal Expansion Process in Epoxy Resin Using a TMA	Thermal Analysis	25
Mechanical Characteristics – Static Strength	Evaluation of Mechanical Characteristics of Various Plastic Products Using Autograph	Autograph	26
Mechanical Characteristics Fatigue and Impact Strength	Evaluation of Endurance (Fatigue Strength) of Various Plastic Products Using Servopulser	Servopulser/Hydroshot	27
Non-Destructive Inspection Defect Inspection and Internal Structural Analysis	Example of Analyzing and Observing the Internal Structure of Various Plastic Products Using Industrial X-Ray Inspection	Industrial X-Ray Inspection System	28
Analysis of Foreign Substances on Film Using an Infrared Microscope	Analysis of Foreign Substances	FTIR	29

Evaluation of Plastic Materials

Evaluation of Raw Materials

Product Evaluation

Product Information

Product Evaluation

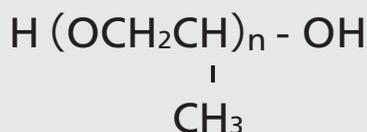
Analysis purpose	Application	Instrument	Page
Hazardous Substances Hazardous Metals	Analysis of Cadmium in Plastics Using an AA Spectrophotometer	AA	30
Hazardous Substances Hazardous Metals	Analysis of Hazardous Metals in Plastics Using an ICP-AES System	ICP-AES	31
Hazardous Substances Hazardous Metals	Analysis of Hexavalent Chromium in Plastic Using a UV-VIS Spectrophotometer	UV	32
Hazardous Substances Organic Substances	Analysis of Phthalate Esters in Plastic	GC/MS	33
Analysis of Hazardous Elements	Analysis of Hazardous Elements in Plastic Using an EDX Spectrometer	EDX	34
Halogen-Free Analysis	Analysis of Halogen Elements (Chlorine) Using an EDX Spectrometer	EDX	35

Product Information

Product	Model	Page
Fourier Transform Infrared Spectrophotometer	IRAffinity-1, IRPrestige-21	36
Single Nano Particle Size Analyzer	IG-1000	37
High-Performance Liquid Chromatograph	Prominence	38
Gas Chromatograph Mass Spectrometer	GCMS-QP2010 Ultra	39
Liquid Chromatograph Mass Spectrometer	LCMS-2020, LCMS-IT-TOF	40-41
MALDI-TOFMS	AXIMA Performance	42
Thermal Analyzer	DSC-60, TMA-60, DTG-60	43
Gas Chromatograph	GC-2014, GC-2010 Plus	44
Scanning Probe Microscope	SPM-9700, WET-SPM Series	45
Infrared Microscope System	AIM-8800	46
UV-VIS Infrared Spectrophotometer	SolidSpec-3700 (DUV), UV-2600/2700, UVmini-1240	47
Autograph	Autograph AG-X Series	48
Servopulser / Hydroshot	Tabletop Servopulser, Hydroshot HITS-T10	49
Industrial X-Ray Inspection System	inspeXio SMX-90CT, inspeXio SMX-225CT	50
Atomic Absorption Spectrophotometer	AA-7000, AA-7000G	51
Multitype ICP Emission Spectrometer	ICPE-9000	52
Energy Dispersive X-Ray Fluorescence Spectrometer	EDX-720	53

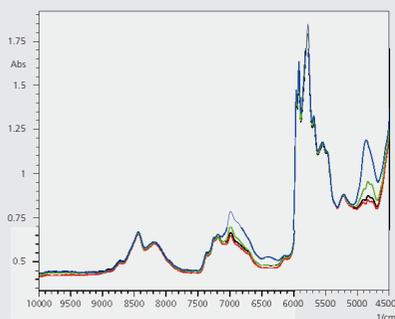
Determination of Hydroxyl Value in Polypropylene Glycol by NIR-PLS Method

Polypropylene glycol (PPG) is a liquid manufactured by polymerizing propylene glycol and is used as a raw ingredient of polyurethane. The hydroxyl value is an index of the content of free hydroxyl groups in polyols, such as PPG. JIS K 1557 (Plastics Polyols for use in the production of polyurethane) specifies using titration to measure the hydroxyl value. However, titration requires equipment and reagents, and is also time-consuming. In contrast, the NIR-PLS (near infrared-partial least squares) method allows calculating the hydroxyl value easily and quickly by simply measuring the near infrared spectrum of the sample, then using PLS to calculate quantities.

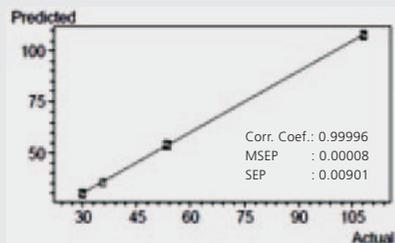


Structural Formula of Polypropylene Glycol

NIR spectra for four homologous types of PPG with different hydroxyl values and a calibration curve created using the PLS method are shown below. The NIR spectra indicate a difference in their peak intensities near 6990 cm^{-1} and 4850 cm^{-1} . The peaks near 6990 cm^{-1} correspond to the first overtone of the O-H stretching vibration, whereas the peaks near 4850 cm^{-1} correspond to a combination of the O-H stretching and bending vibrations.



NIR Spectra of Polypropylene Glycol



Actual Hydroxyl Value (known concentration) vs. Predicted Hydroxyl Value (quantitative value)

NIR Heating Transmission Cell

This cell allows measuring transmittance while liquid samples placed in the provided 6-mm diameter test tube are heated or held at a constant temperature. This is especially convenient for quantitative analysis of multiple components in liquid samples or when monitoring reactions during heating processes.

Specifications

- Measurement range : 12,500 to 3,800 cm^{-1}
- Temperature range : Room temp. to 120°C
- Accessory recognition function : Included
- Test tube : 6 mm dia. × 50 mm



NIR Heating Transmission Cell

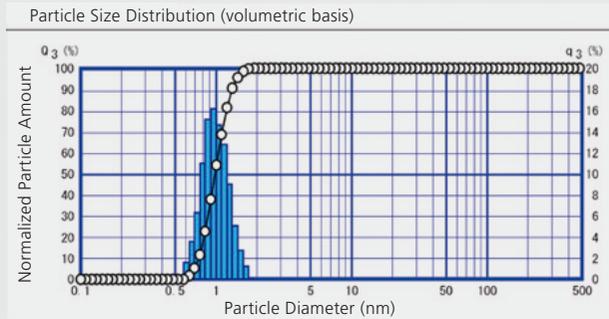
Particle Size Control and Particle Size Distribution

High-Sensitivity, High-Reproducibility Measurement of Single-Digit Nanoparticles Dispersed in Liquid

Example of Measuring Fullerene Hydroxide

Even fullerene hydroxide particle sizes, which typify samples in the single-digit nanometer region, can be measured with high sensitivity and good reproducibility. This enables accurately evaluating the dispersivity of particles in the single nanometer region.

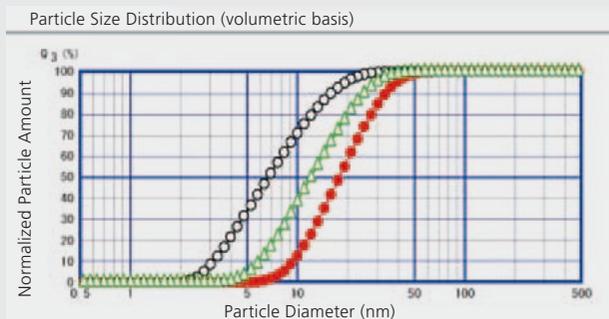
Sample provided by Professor Kokubo of Osaka University



Example of Measuring Mixed Samples (colloidal silica)

- A (only)
- B (only)
- △ A + B (mixture)

Even mixed samples can be measured. The IG method utilizes the diffusion of a diffraction grating formed by particles to measure samples, so the signal level is not dependent on particle size. Therefore, this allows evaluating mixed samples. In contrast, evaluating mixtures is difficult with methods that use scattered light, because the signal level is proportional to the cube of the particle diameter, even if the volume is the same.



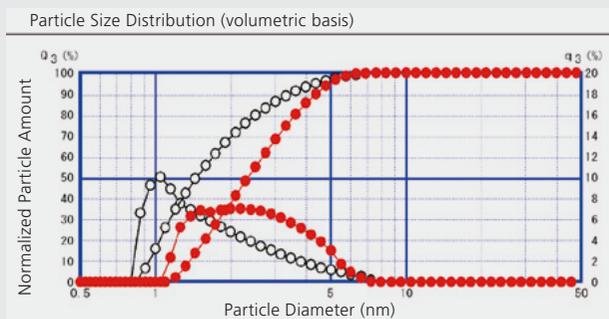
Example of Measuring Zinc Sulfide (ZnS)

This example compares IG-1000 and TEM measurement results (mean particle diameter).

These show a very consistent relationship between sizes.

	IG-1000	TEM
○	1.63	1.98
●	2.39	2.22

Samples provided by Professor Mori of the Doshisha University



Example of Recycling Preparative Separation of Polystyrene

HPLC is widely used as a means to purify samples by separation, but the large preparative columns become quite expensive. Any cost limitations affect column length and quantity to achieve better separation. In such situations, separation can be improved by recycling the eluent band containing the target components through the column several times to effectively extend the column length. In this example, a Prominence **semi-preparative recycling system** was used to separate polystyrene by GPC.

Fig. 1 illustrates the flow line of the Prominence **semi-preparative recycling system**. The sample is loaded by autosampler or manual injector, then separated by the preparative column and eluted to the detector. Here, the system uses a recycle valve to divert the eluate either to the flow line leading to a fraction collector, or to the flow line leading back to the pump inlet. By switching the recycle valve to divert the flow back to the pump inlet when target peaks are eluted, the sample can be recycled through the preparative column as many times as necessary to obtain a separation equivalent to using a longer column. Since recycling the eluate does not consume any new mobile phase, it also helps reduce solvent consumption.

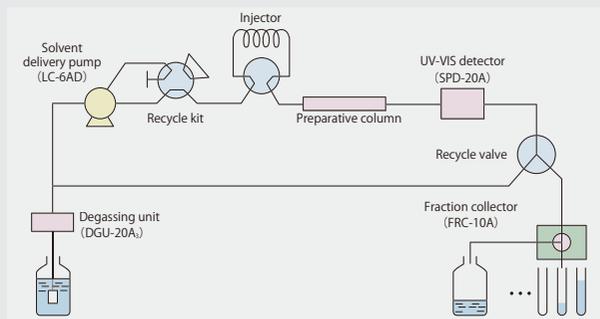


Fig. 1 Flow Line of Prominence **Semi-Preparative Recycling System**

Fig. 2 shows an example of a recycle separation of polystyrene molecular weight markers (molecular weight = 781 and polydispersity* < 1.15). Portions indicated by arrows are recycled. This shows how even polystyrene, which has low polydispersity, can be separated into many oligomer peaks. By switching the recycle valve to the flow line leading to the FRC, target peaks are easily collected.

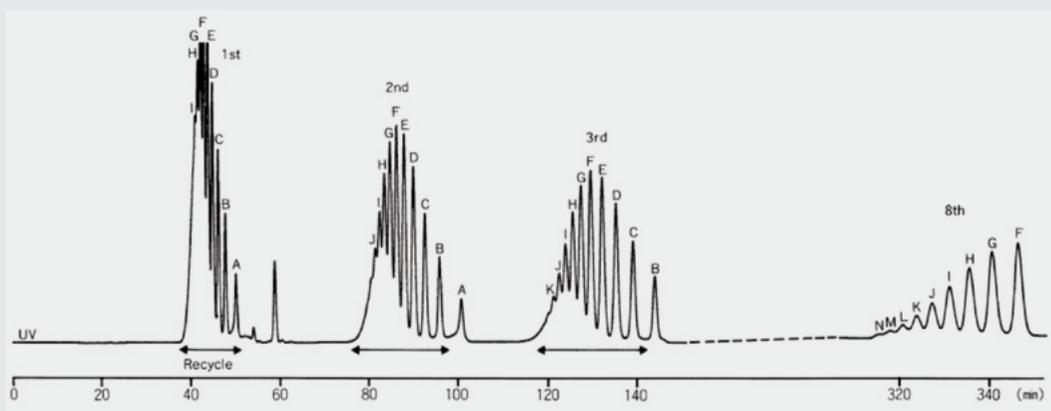


Fig. 2 Example of Recycling Separation of Polystyrene

Analysis Conditions

Instrument : Prominence **semi-preparative recycling system**
 Analytical Column : Shim-pack GPC-20025C+2002C+2001C
 (each 300 mmL × 20 mm I.D.)
 Mobile Phase : Chloroform

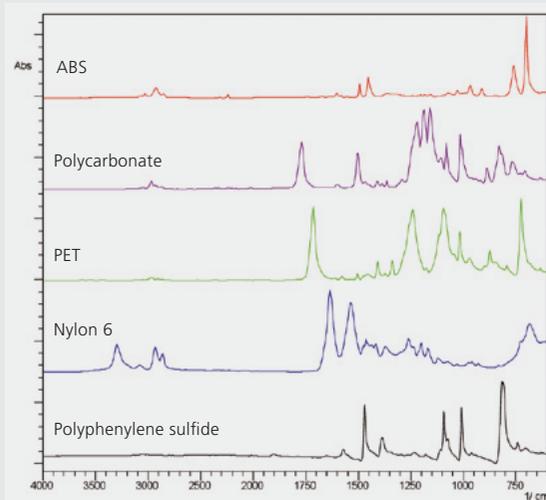
Flowrate : 3.0 mL/min
 Column Temperature : Ambient
 Detection : UV 254 nm

* Polydispersity = Weight-Average Molecular Weight / Number-Average Molecular Weight

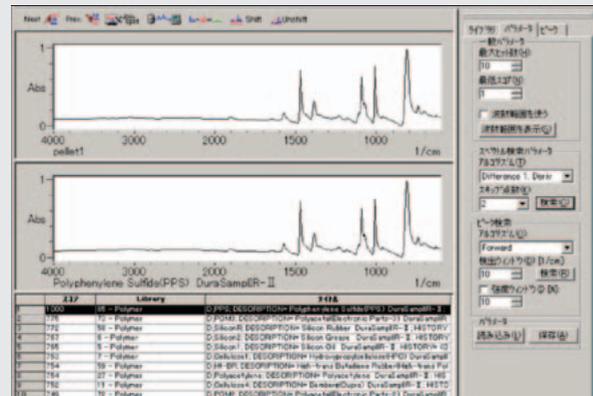
Structure and Composition

FTIR Measurement of Plastic Pellets and Powder by Single Reflection ATR Method

The figure on the left below shows results from measuring various types of plastic pellets and powders. The single reflection ATR method allows measuring pellet and powder samples directly by simply placing them tightly against an approximately 2 mm diameter prism. Furthermore, it allows even new users to easily obtain qualitative information about measured polymers by using the spectral search function.



Example of Measuring Plastic Pellets and Powders



Spectral Search Window
Example of Measuring Plastic Pellets and Powders

Single Reflectance ATR Method

Single reflection ATR measures the infrared spectrum of a sample pressed against the surface of a prism approximately 2 mm in diameter. It allows measuring samples by simply placing them in close contact with the center of the prism. Therefore, it is not restricted to flat samples and can measure curved or powdered samples as well. Liquid samples can be measured by simply dripping them onto the prism. A photograph of the DuraSamplIR II single reflection ATR attachment and an illustration of its optics are shown below. It irradiates the sample with infrared light through the prism and obtains information about the sample surface (to a depth of about 1 μm) based on the light reflected from the boundary surface. Not only does this eliminate the need for dilution or other sample pretreatment, but also allows obtaining information about the sample surface easily.



Photograph of DuraSamplIR II

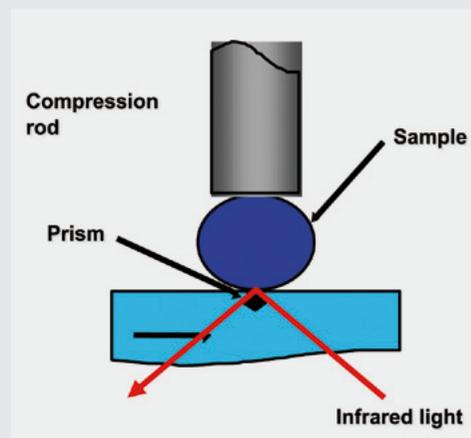


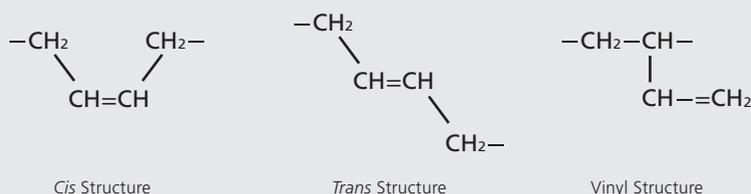
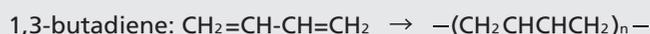
Diagram of Optics

Structure and Isomers

FTIR

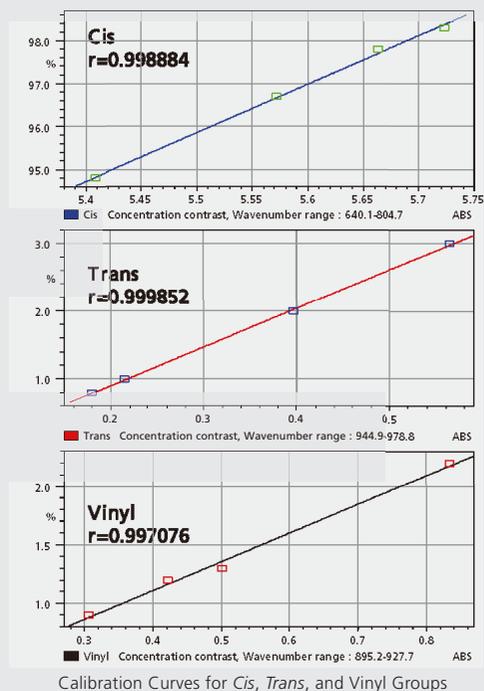
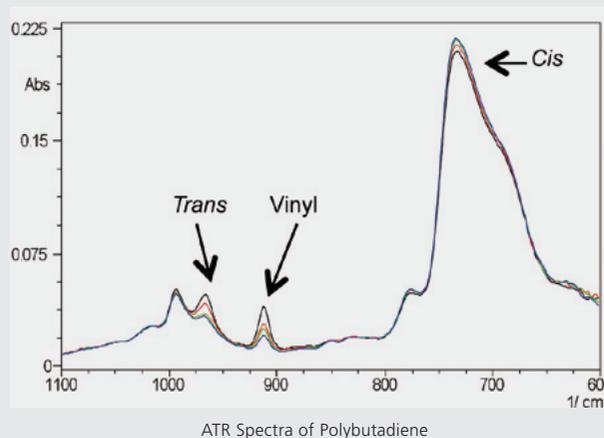
FTIR Analysis of Polybutadiene Microstructure Using Single Reflection ATR

Polybutadiene is a polymer with characteristics of both plastic and rubber. It is a collective term for multiple polymer configurations of 1,3-butadiene monomers. These can polymerize into three types of structures, the *cis* structure (*cis*-1,4-polybutadiene), the *trans* structure (*trans*-1,4-polybutadiene), and the vinyl structure (1,2-polybutadiene). Of these structures, polybutadienes containing larger amounts of the plastic-like vinyl structure are used for applications such as wrapping films, whereas polybutadienes containing larger concentrations of the rubber-like *cis* structure are used for rubber products, such as tires and belts.



The proportion of *cis*, *trans*, and vinyl structures in a polybutadiene can be determined using NMR spectra, infrared spectra, and other methods. The Morero method using infrared spectra is considered useful if the polymer contains a high proportion of *cis* structures. The Morero method involves dissolving the sample in carbon disulfide and measuring the transmittance using a fixed cell with 1 mm optical path length. Then the proportion of each structure is determined from the absorbance of the *cis*, *trans*, and vinyl group peaks. Since this method uses transmittance measured using a fixed cell, it provides good sensitivity and enables using dispersive IR systems for quantitation. However, this method requires weighing and dissolving samples, cleaning cells, and other processing. It also requires a significant amount of carbon disulfide. In contrast, the ATR method allows measuring samples directly, without requiring any pretreatment, and the prism can be cleaned with a tiny amount of alcohol or other cleaner.

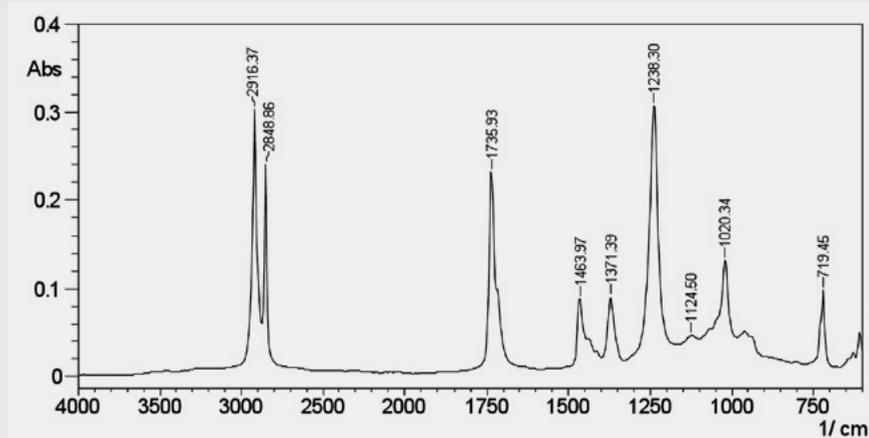
Shown below are spectra of polybutadiene with 90% or higher *cis* content obtained using a DuraSAMPLR II single reflection ATR attachment. The peaks near 733 cm^{-1} , 912 cm^{-1} , and 966 cm^{-1} correspond to *cis*, vinyl, and *trans* groups, respectively. Calibration curves for each group are shown to the right, all of which show good linearity.



Structure and Copolymers

Quantitation of Vinyl Acetate in EVA

The copolymer ethylene-vinyl acetate (EVA) offers good clarity and is used for a wide variety of products, such as fertilizer bags, wrapping film, bottles, and tubes. Increasing the content of vinyl acetate in EVA increases its elasticity and thermal plasticity. Though there are several methods available for quantitating the content of vinyl acetate in EVA, infrared spectrophotometry is easier and faster because analysis can be performed without pretreating samples.

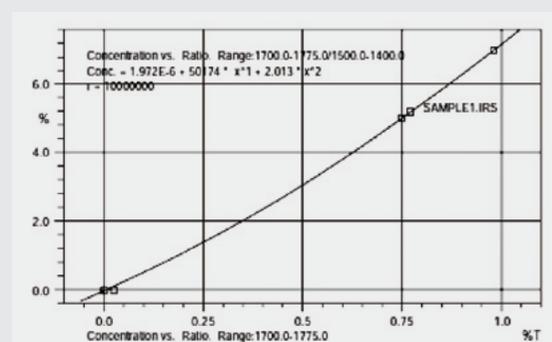
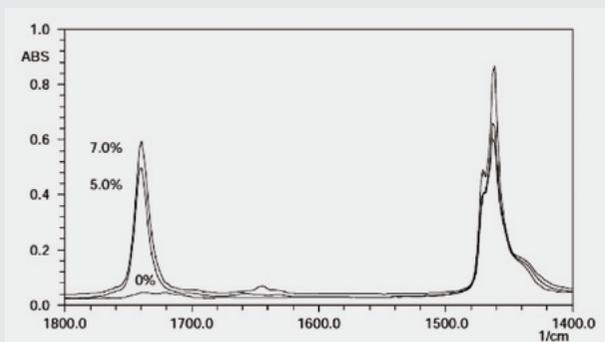


The EVA spectrum above was measured using the ATR method. The peaks near 1736 cm^{-1} and 1238 cm^{-1} correspond to C=O and C-O bond stretching vibration of vinyl acetate. The peaks near 2916 cm^{-1} , 2849 cm^{-1} , and 1464 cm^{-1} are primarily due to -CH₂ in ethylene.

The results shown below on the left (enlarged view) are from samples containing 0%, 5%, and 7% vinyl acetate, measured using a horizontal ATR attachment. The show that the peak intensity near 1736 cm^{-1} increases with increasing concentrations of vinyl acetate.

The calibration curve below on the right was created using the intensity ratio of peaks near 1736 cm^{-1} and 1464 cm^{-1} .

In this way, the concentration of vinyl acetate in EVA can be determined from the peak intensities in infrared spectra.



Analysis of Additives in Plastic

Polymer materials are often formulated using different types of additives depending on the grade or manufacturer. Analyzing a sample extract generally requires separating the additives from the polymers. Conventionally, they are separated by solvent extraction or precipitation methods, but that requires time-consuming pretreatment, including thoroughly washing the equipment used for pretreatment. The thermal desorption GC/MS method described below improves productivity by facilitating quick analysis and utilizing disposable pretreatment equipment (ECO cup).

Instrument Configuration

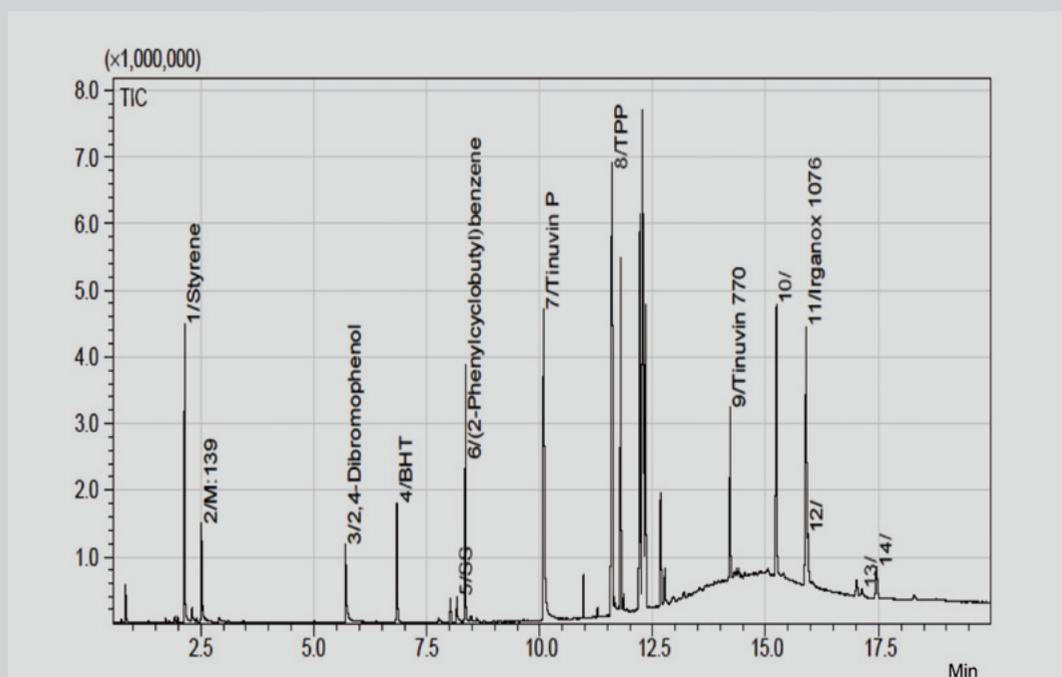
GCMS-QP2010 Ultra

Pyrolysis System: EGA/PY-3030D

(from Frontier Laboratories, Ltd.)



Thermal Extraction of PS Plastic Shavings
(1 mg) for 5 Minutes at 300°C



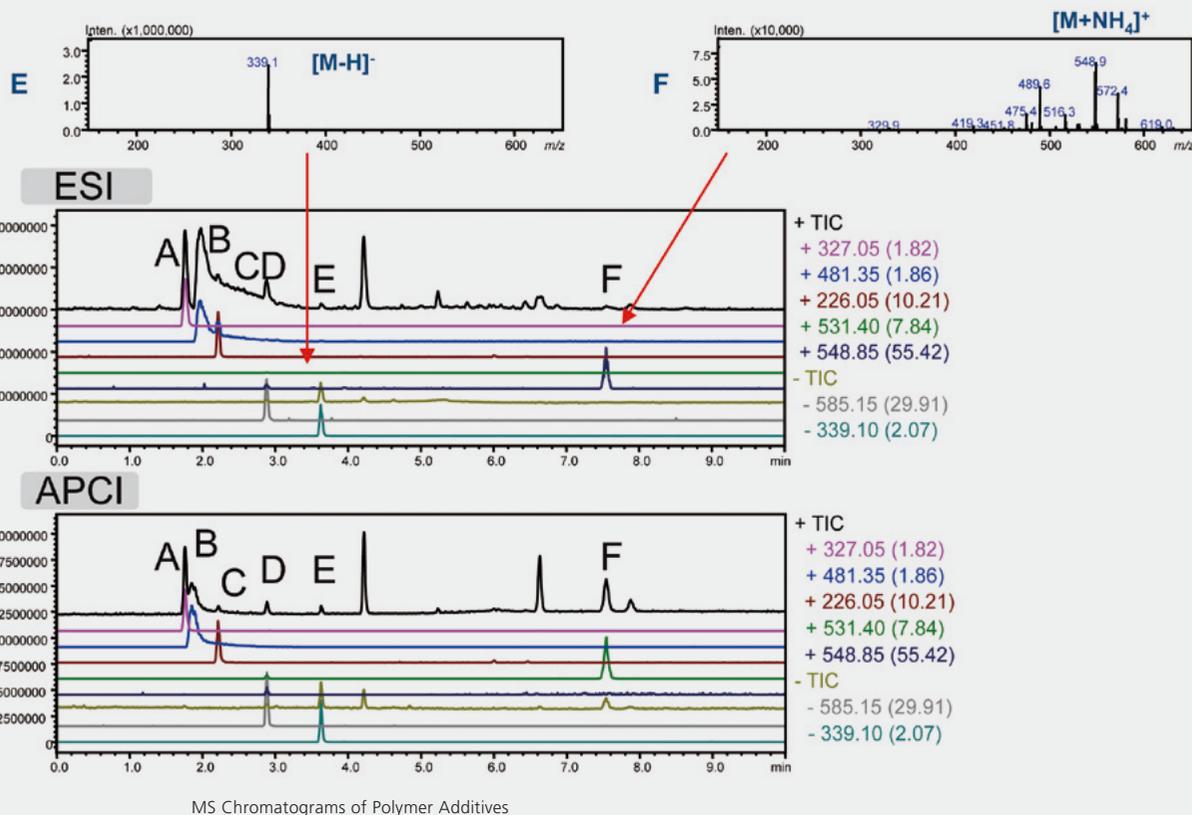
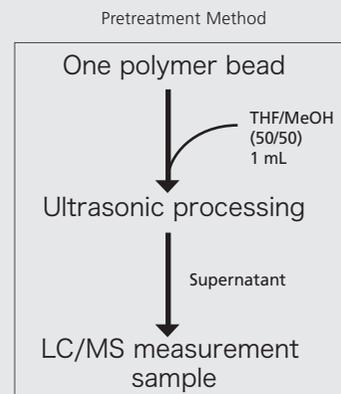
Analysis of Evolved Gas from PS Plastic

Analysis of Additives in Plastic Using LCMS-2020

An organic solvent (in this case, THF/MeOH = 1/1) was added to a known quantity of a plastic sample. It was then sonicated and the supernatant was extracted and directly analyzed. The sample was ionized by ESI and APCI, and was monitored simultaneously in both positive and negative modes using high-speed polarity switching. Of the six types of additives observed, components D and E were detected with higher sensitivity in negative mode. Simultaneous monitoring in both modes provides sensitive and accurate data, because the optimal ionization parameters can be applied to each compound.



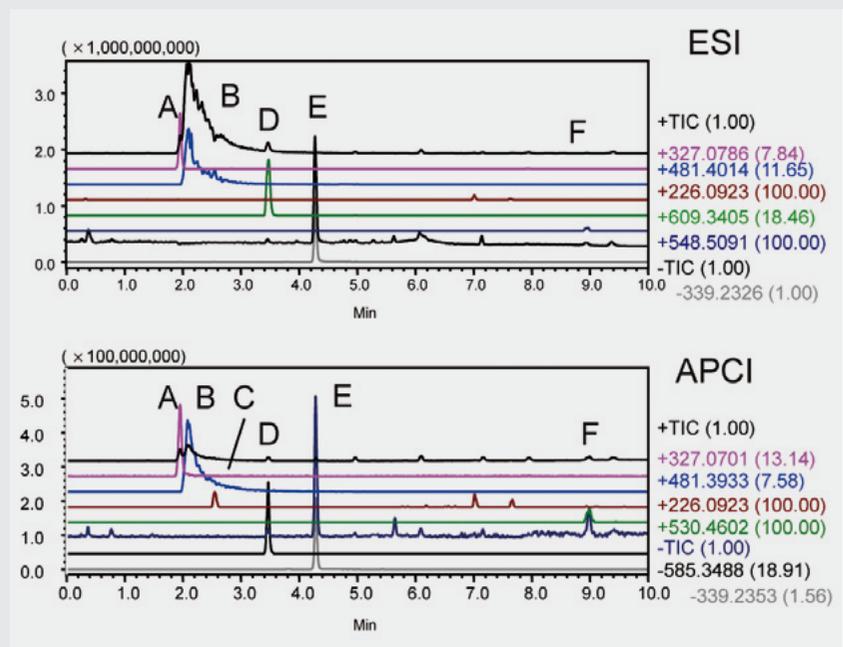
Polymer beads used in the analysis



Additives

LCMS-IT-TOF

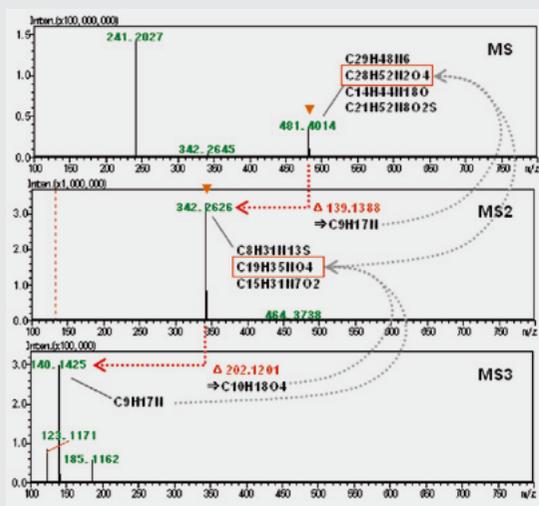
Analysis of Additives in Polymer Extract Using LCMS-IT-TOF (Part 1)



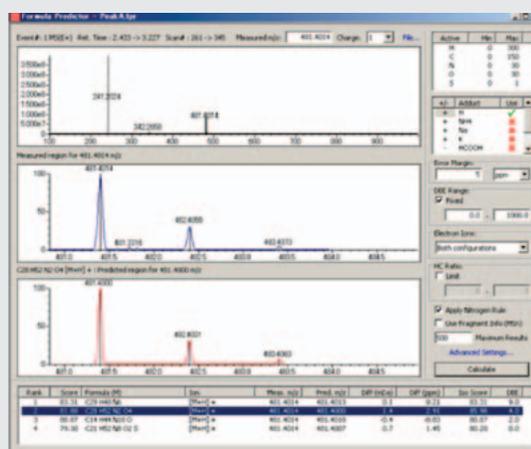
An organic solvent (in this case, THF/MeOH = 1/1) was added to a known quantity of polymer beads. The samples were then sonicated and the supernatant was extracted and directly analyzed by an LCMS-IT-TOF system. Measurement results are shown in the chromatograms on the left.

Of the six types of additive peaks observed, the below structural prediction was performed for peak B.

Example: Predicting the Structure for Peak B

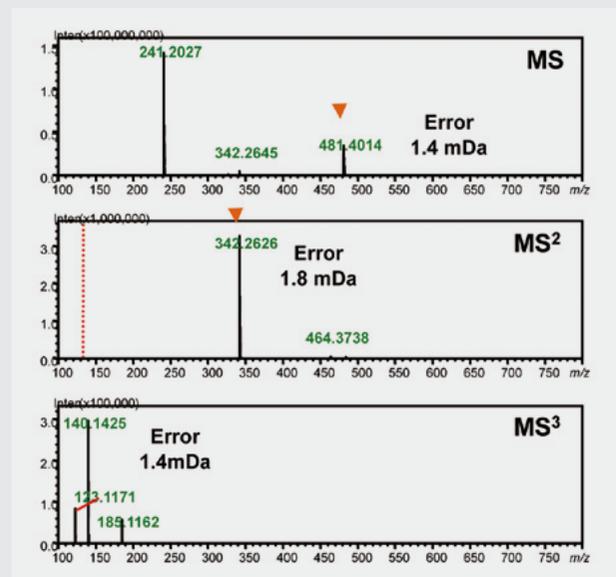


Example: Analysis Using Formula Predictor



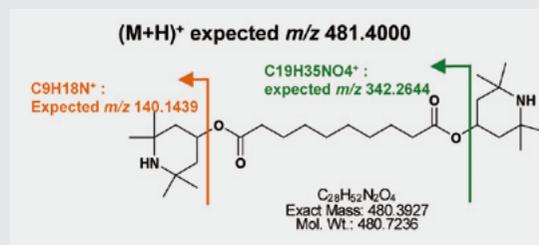
The smaller the m/z number, the fewer the number of candidates. Using MS^n analysis allowed the structural predictions to be narrowed down.

Analysis of Additives in Polymer Extract Using LCMS-IT-TOF (Part 2)

– Predicting the Structure of Additives and Attributing MS³ Measurement Results –

An organic solvent (in this case, THF/MeOH = 1/1) was added to a known quantity of polymer beads. Then they were processed in a sonicator and the supernatant was taken as the measurement sample and measured by an LCMS-IT-TOF system. The composition was then predicted for one of the six types of additive peaks observed. This resulted in obtaining a predicted formula (C₂₈H₅₂N₂O₄). (For more details regarding the composition prediction process, refer to Evaluation of Plastic Materials – 7A. Additives in the Plastic Analysis Catalog.) Searching for this formula on the Internet revealed that it is likely to be decanedioic acid bis (2,2,6,6-tetramethyl-4-piperidyl) ester, an additive generally used as a stabilizer.

Ions detected in the MS³ measurement were then attributed to the predicted compound. This confirmed that the detected ions suggested the predicted structure, with minimal error for each product ion.



Prediction Results for Each Peak

(For more details regarding the composition prediction process, refer to Evaluation of Plastic Materials – 7A. Additives in the Plastic Analysis Catalog.)

	Retention Time (min)	Name of Predicted Compound	Composition Formula (Molecular Weight)	m/z		Error
				Calculated Value	Measured Value	
A	1.95	Triphenyl phosphate	C ₁₈ H ₁₅ O ₄ P (326.0708)	327.0781 ([M+H] ⁺)	327.0791 ([M+H] ⁺)	1.0 mDa 3.1 ppm
B	2.10	Decanedioic acid bis (2,2,6,6-tetramethyl-4-piperidyl)ester	C ₂₈ H ₅₂ N ₂ O ₄ (480.3927)	481.4000 ([M+H] ⁺)	481.4014 ([M+H] ⁺)	1.4 mDa 2.9 ppm
C	2.55	Tinuvin P	C ₁₃ H ₁₁ N ₃ O (225.0902)	226.0975 ([M+H] ⁺)	226.0972 ([M+H] ⁺)	0.3 mDa 1.3 ppm
D	3.47	Onchitriol II A	C ₃₄ H ₅₀ O ₈ (586.3506)	585.3433 ([M-H] ⁻)	585.3435 ([M-H] ⁻)	0.2 mDa 0.33 ppm
E	4.29	2,2-Bis(3-sec-butyl-4-hydroxyphenyl)propane	C ₂₃ H ₃₂ O ₂ (340.2402)	339.2330 ([M-H] ⁻)	339.2325 ([M-H] ⁻)	0.5 mDa 1.4 ppm
F	8.94	Irganox 1076	C ₃₅ H ₆₂ O ₃ (530.4699)	548.5037 ([M+NH ₄] ⁺)	548.5049 ([M+NH ₄] ⁺)	1.2 mDa 2.2 ppm

Analysis of Additives in Acrylic Sheets

Polymer materials degrade due to a variety of factors, including light, heat, mechanical and electrical factors, radiation, chemicals, and moisture. Many different additives, such as antioxidants, UV absorbers, hindered amine light stabilizers (HALS), and thermal stabilizers, have been developed to prevent such degradation and maintain the desired characteristics of the polymer material. The quantity of each additive used varies depending on the specific purpose of the polymer material, but generally is in **ultra trace quantities of about 1% (w/w) or less**. Even for the same polymer material, the type of additive can vary depending on the grade or manufacturer. Consequently, analyzing the additives in polymer materials is extremely important for investigating or improving the performance of various polymer materials.

In this example, an SEC-AccuSpot-AXIMA system is used to detect the additives in a commercial acrylic sheet product. The measurement sample was prepared by cutting about 1 g from a commercial acrylic sheet and sonicating it immersed in 1 mL of tetrahydrofuran (THF) for about 1 minute. The supernatant was then used as the measurement sample (Fig. 1). Fig. 2 shows SEC chromatograms for the samples extracted from the acrylic sheet. The AccuSpot system was used to collect fractions at six second intervals, from the time the primary peaks started eluting until they finished (7 minutes to 17 minutes 30 seconds), resulting in 105 sample spots on a MALDI sample plate.

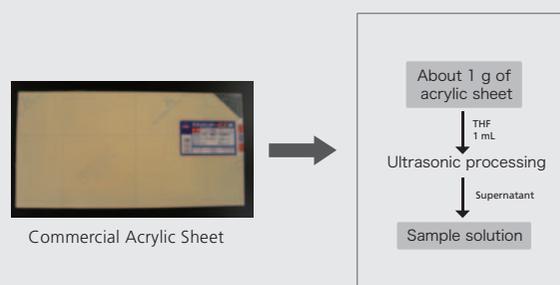


Fig. 1 Sample Preparation

Analytical Conditions

- **SEC** Column : Shodex GF310A-1E (1.0 mmI.D. x250 mm)
Flowrate: 10 μ L/min; Eluent: THF
Detector: UV (λ = 220 nm); Injection volume: 1 μ L
- **AccuSpot** Spot interval: 6 sec; Loadage: 1 μ L/well
Loadage (mixture of matrix and cationizing reagent): 0.2 μ L/well
- **MALDI-TOFMS** Matrix: Dithranol-20 mg/mL- THF
Cationizing reagent: Na-TFA-10 mg/mL-THF

Each spot was measured using a Matrix Assisted Laser Desorption Time of Flight Mass Spectrometer (MALDI-TOFMS) to obtain mass spectra (Fig. 2) corresponding to elution times. The fraction collected after 11 minutes 10 seconds shows a peak detected for the additive [IRGANOX 1010 + Na]⁺ at m/z 1200, which was not visible before separation in the mass spectrum, even after magnifying by five times (Fig. 3). In addition to the additives, three different types of molecular weight distributions were detected (● □ ▲), with a peak near m/z 2000.

Since in each case, the interval between neighboring peaks is 100, these molecular weight distributions are presumably all due to oligomers consisting of identical monomer units.

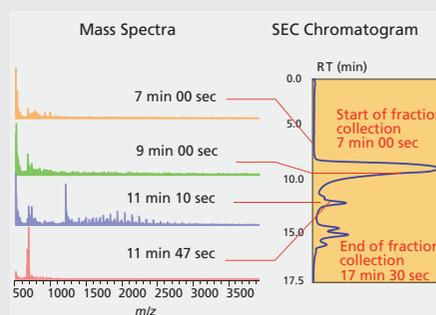


Fig. 2 Mass Spectra of Respective Fractions

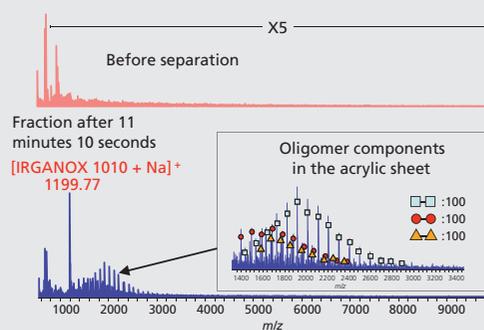
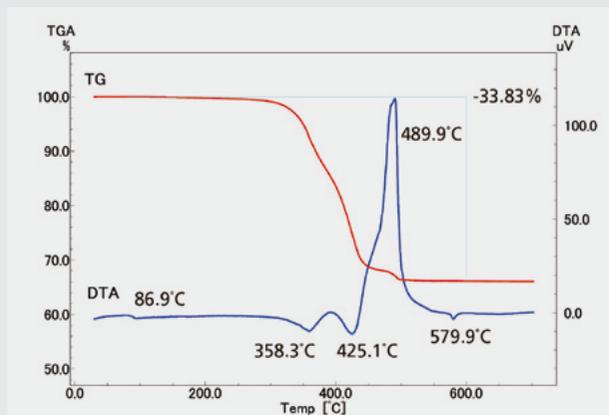


Fig. 3 Detection of Additives in an Acrylic Sheet

Quantitation of Reinforcing Materials in Epoxy Resins

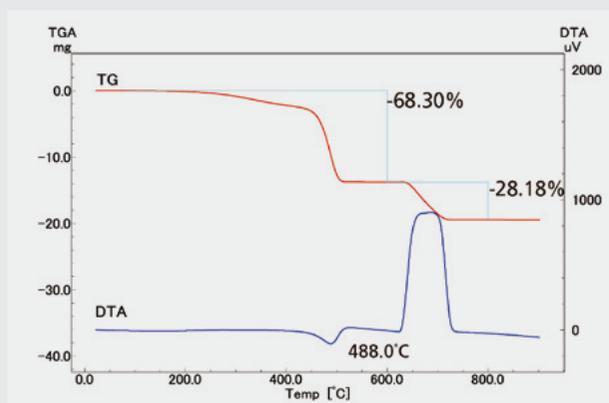


Quartz and other reinforcing materials in plastics are quantitated based on the quantity remaining after heating the sample in air until the resin components are completely combusted. In this example, the quantity of quartz in epoxy resin is quantitatively analyzed. When the sample was heated in air, the epoxy resin was completely combusted by 600°C and the weight decreased by 33.83%.

The concentration of quartz was calculated as $100 - 33.83 = 66.17\%$.

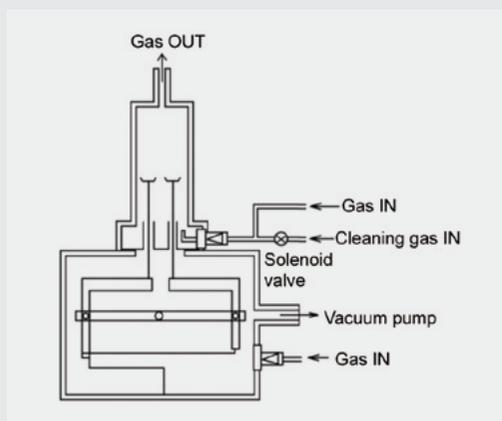
On the DTA curve, the transition of quartz was measured at 580°C, along with the endothermic and exothermic peaks due to decomposition and combustion of the epoxy resin between 300 and 600°C.

Quantitation of Carbon Black in SBR



The quantity of carbon black in resin is measured by heating the resin and switching from a nitrogen atmosphere to air. In the figure to the left, the amount of carbon black contained in SBR was quantitated. First the SBR was heated in a nitrogen atmosphere to decompose all components of the rubber other than carbon black. After completely decomposing the SBR by heating to about 600°C, the atmosphere was switched to air. When the carbon black combusted in air, the total decrease in weight was observed and the carbon content was quantitated from the percentage decrease. In the figure to the left, this resulted in a quantitated value of 28.2%. Note, however, that combusting carbon black generates residual inorganic matter.

Configuration of DTG Gas Flow Lines



Due to its unique gas flow line configuration, the Shimadzu DTG-60 thermal analyzer can be used for a wide variety of measurement applications. For measurements in inert gas, the vacuum pump exhaust is utilized to displace the gas quickly. The gas inlet is located near the sample, so that it is also possible to measure samples with active gas flowing only to the sample area, while keeping the separate balance area protected with inert gas.

Analysis of Trace Homopolymer Components in Copolymers

The following is an example of using an SEC-AccuSpot-AXIMA system to analyze trace homopolymer components in samples, based on a model sample consisting of the binary copolymer poly(methyl methacrylate-*b*-*n*-butylmethacrylate) (poly(MMA-*b*-*n*-BMA)). A microscale separation column was used to load all components onto MALDI sample plates without any sample loss. Fig. 1 shows the SEC chromatogram of poly(MMA-*b*-*n*-BMA). The AccuSpot system was used to collect fractions at six second intervals, from the time the primary peaks started eluting until they finished (for 5 minutes, from 8 minutes 20 seconds to 13 minutes 20 seconds), and load 50 sample spots onto a MALDI sample plate. Each spot was measured in a MALDI-TOFMS system to obtain mass spectra (Fig. 2) corresponding to elution times. The mass spectrum obtained from each fraction indicates a different molecular weight distribution.

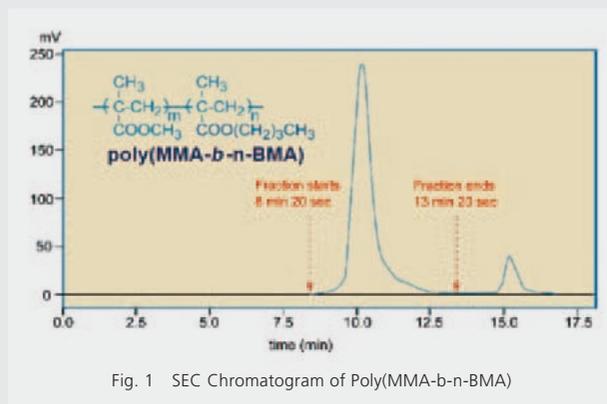


Fig. 1 SEC Chromatogram of Poly(MMA-*b*-*n*-BMA)

Analytical Conditions

- **SEC** Column: Shodex GF310A-1E (1.0 mm I.D. × 250 mm)
Flowrate: 10 μ L/min; Eluent: THF
Detector: UV ($\lambda = 220$ nm); Injection volume: 1 μ L
- **AccuSpot** Spot interval: 6 sec, 1 μ L/well
Mixture of matrix and cationizing reagent: 0.2 μ L/well
- **MALDI-TOFMS** Matrix: Dithranol-20 mg/mL- THF
Cationizing reagent: Na-TFA-10 mg/mL -THF

The fraction collected after 11 minutes and 18 seconds shows a detected molecular weight distribution not observed before separation (Fig. 3). The 142 interval between adjacent peaks indicates that the molecular weight distribution with a peak near m/z 1200 (marked with ●) corresponds to the PnBMA homopolymer. Similarly, the 100 interval between adjacent peaks indicates that the molecular weight distribution with a peak near 1800 (marked with ■) corresponds to the PMMA homopolymer. These results indicate that the poly(MMA-*b*-*n*-BMA) in this example, contains trace amounts of the homopolymer components PnBMA and PMMA.

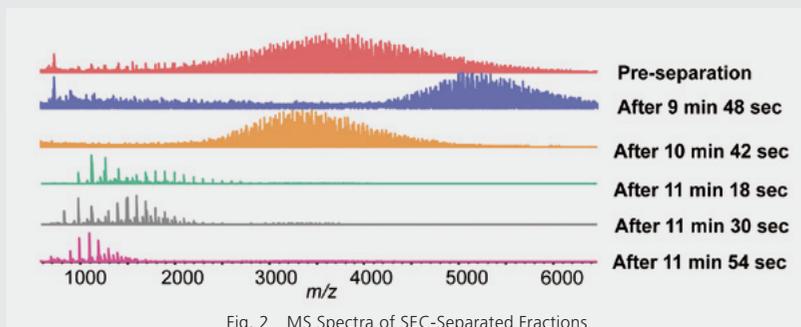


Fig. 2 MS Spectra of SEC-Separated Fractions

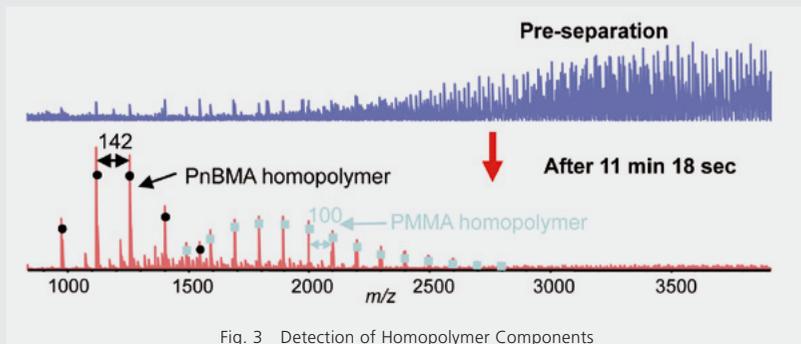


Fig. 3 Detection of Homopolymer Components

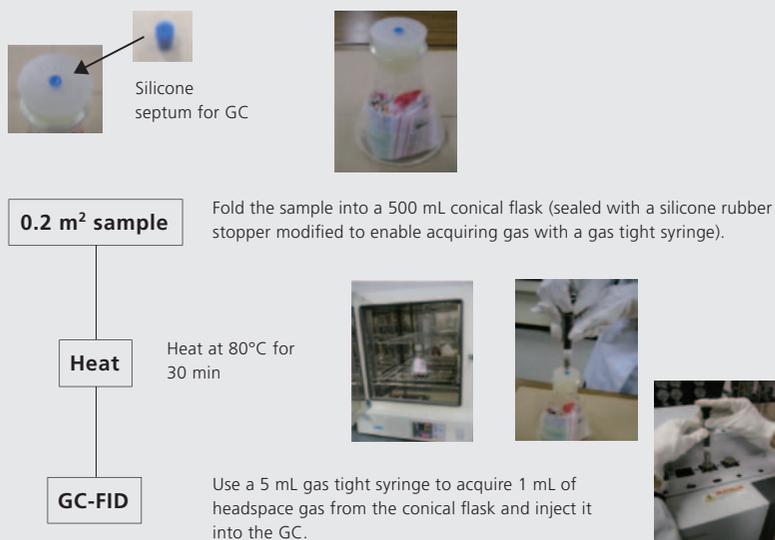
Analysis of Residual Organic Solvents in Flexible Packaging Materials

With such a high interest in security, safety, and health in recent years, measuring residual organic solvents in plastic products is now more important than ever. Residual solvents are commonly analyzed using headspace gas chromatography, which involves placing the sample in a vial and heating it to a given temperature for a given time, and then injecting the resulting gas phase into a gas chromatograph. This allows using simple pretreatment procedures to measure highly volatile components with relatively high sensitivity. Due to the simple pretreatment, samples can be left in the vial for analysis.

Quantitative values from analyzing solids by headspace GC are calculated from the quantity of volatilized components in the gas phase at a given maintained temperature. Therefore, unvolatilized components that remain trapped in the solid sample are not included in quantitative values. Consequently, the method is used to measure and compare the quantity of volatile components evolved from different samples under certain conditions.

In this example, flexible packaging material was analyzed using the residual solvent measurement method (manual HS method) specified in the Control Devices Manual for Manufacturing Flexible Packaging Materials: Flexible Packaging Hygiene Association Edition.

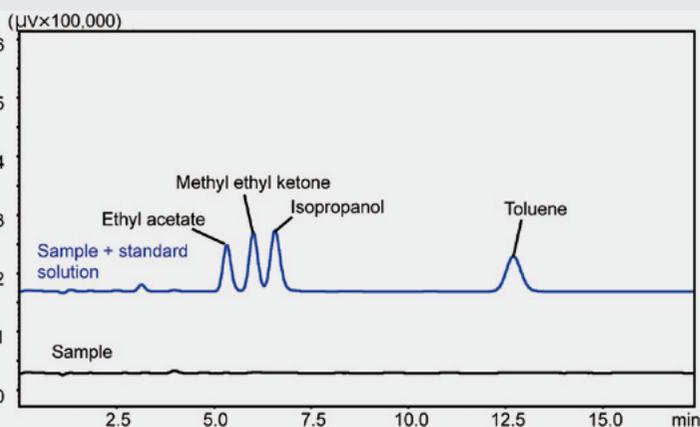
Residual solvent measurement method (manual HS method) specified in the Control Devices Manual for Manufacturing Flexible Packaging Materials: Flexible Packaging Hygiene Association Edition



Features of Headspace Methods

- Able to analyze highly volatile components (substances with low boiling points) with high sensitivity
- Maintenance is easy because non-volatile components never enter the GC unit.
- Liquid samples containing components with high boiling points can be analyzed more quickly than by direct injection.
- Using a fully automatic headspace sampler improves productivity.

This method is not suitable for difficult-to-volatilize components.



Example of Fast Analysis of Residual Organic Solvents in Flexible Packaging Materials

System Configuration GC-2014 + GCsolution



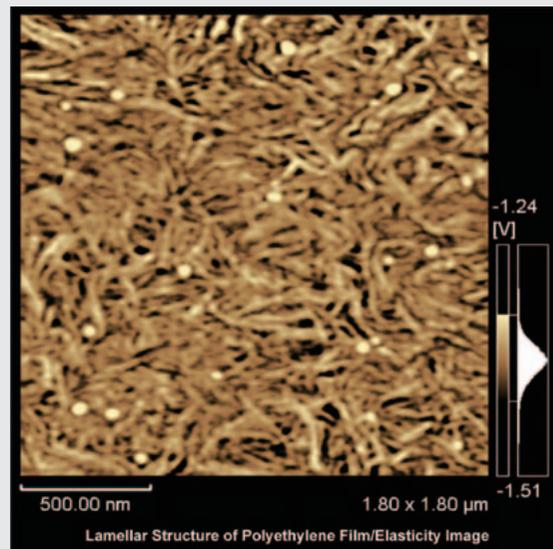
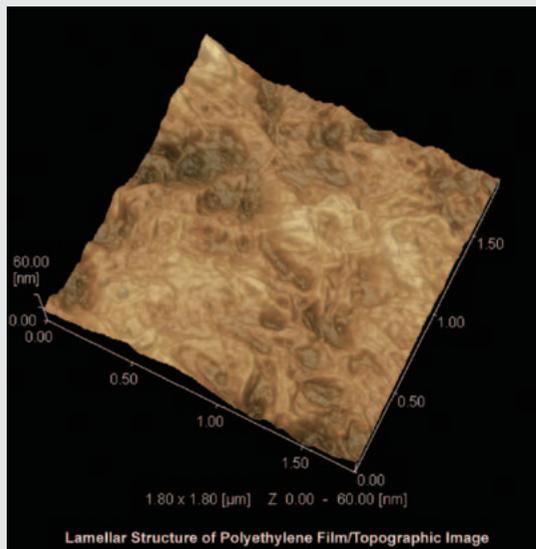
GC-2014 + GCsolution

Surface Structure

SPM

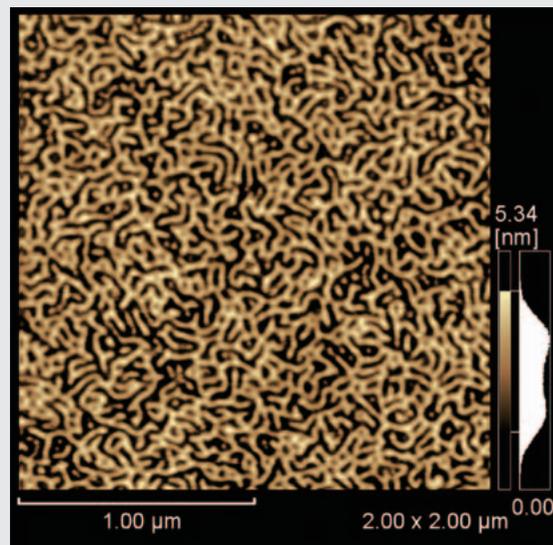
Observing the Lamellar Structure of Polymer Film Using a Scanning Probe Microscope

The surface of polymer films have a characteristic form typified by a lamellar structure that is related to the material's transparency and strength. In this example, the surface of polyethylene film is observed via a scanning probe microscope (SPM) to simultaneously obtain a three-dimensional image (left) and phase image (right) of the surface. The three-dimensional image shows that there are tiny bumps on the surface. The phase image clearly shows the lamellar structure and crystallization of the polymer. The areas where crystallization has progressed and become harder appear brighter. The image is 1.8 μm on each side. In this way, using an SPM allows easily observing the structure and physical properties of polymer films under atmospheric conditions, without any special sample preparation.



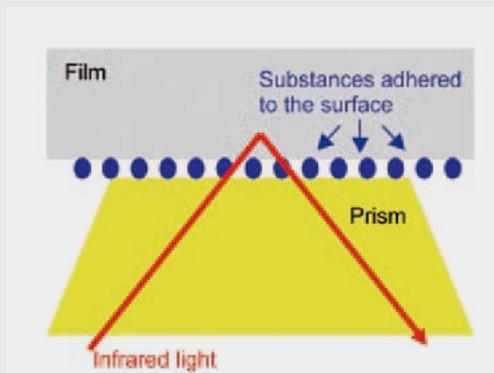
Micro Phase Separation of Block Copolymers Using a Scanning Probe Microscope

A phase separation structure can be observed in plastics that include a mixture of two or more materials. The image to the right is the block copolymer surface observed using a scanning probe microscope (SPM). Due to the SPMs high resolution capability, it clearly shows micro phase separation structures. Due to its ability to observe samples in atmospheric conditions, it is being used for an increasing range of applications, such as to observe the heating, cooling, tension, or cross sections of samples.

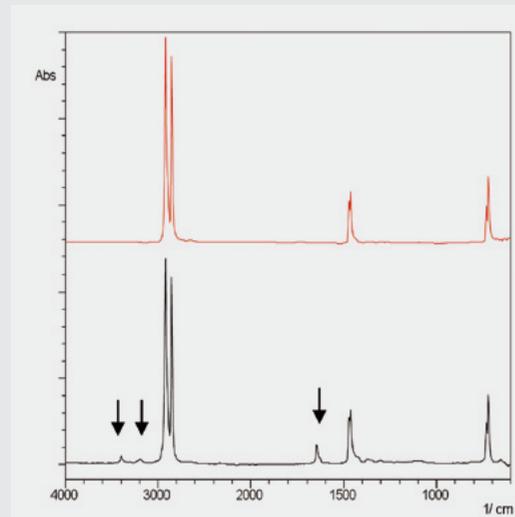


Analysis of Film Surface by FTIR-ATR Method

The ATR (attenuated total reflection) method obtains information about a sample surface held closely against a prism. Therefore, it can be used to obtain information about coating materials, adhesives, or precipitates from within a sample on the sample surfaces, without requiring any sampling or other pretreatment processes. The figures below show the results (black) from measuring a commercial film and ATR spectrum (red) of typical polyethylene sample. This shows that the commercial film product is made of polyethylene, but it also shows peaks that are not observed in the standard polyethylene.



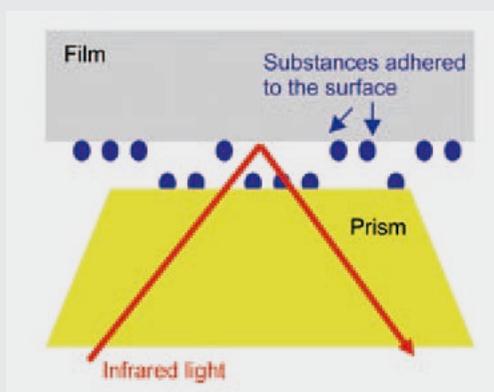
ATR Measurement Optics with Sample Held Tightly Against Prism



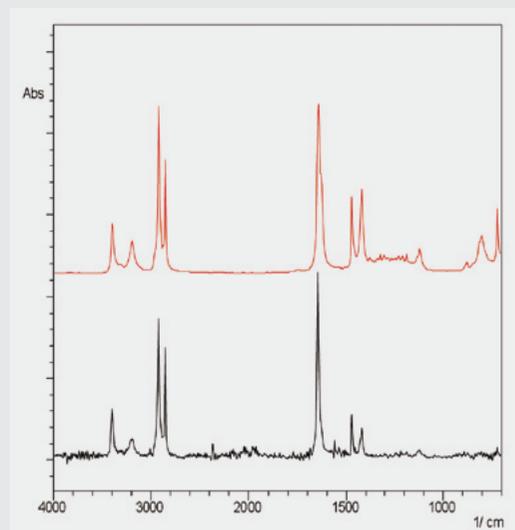
ATR Spectrum of Commercial Film Product (Black) and ATR Spectrum of Polyethylene (Red)

The figure on the right was obtained by removing the commercial film from the prism and measuring only what remained on the prism. Since the ATR method involves pressing the sample against the prism, substances from the sample surface, precipitates from within, or other substances can be transferred to the prism. In such cases, information from only the transferred substance can be obtained by removing the sample and measuring again.

In this case, it was determined that the transferred substance was a fatty acid amide, such as stearic acid amide. Fatty acid amides are commonly used as polyethylene smoothing agents, releasing agents, printing ink additives, or pigment dispersing agents.



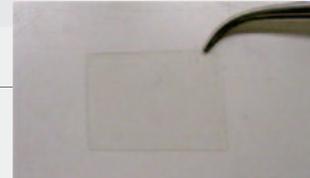
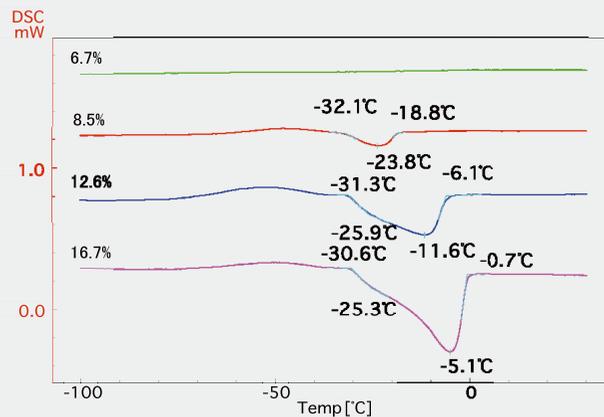
ATR Optics for Measuring Transferred Substances



ATR Spectrum of Transferred Substances (Black) and ATR Spectrum of Stearic Acid Amide (Red)

Measurement of Water Melting in a Polymer Electrolyte Fuel Cell Membrane Using a DSC

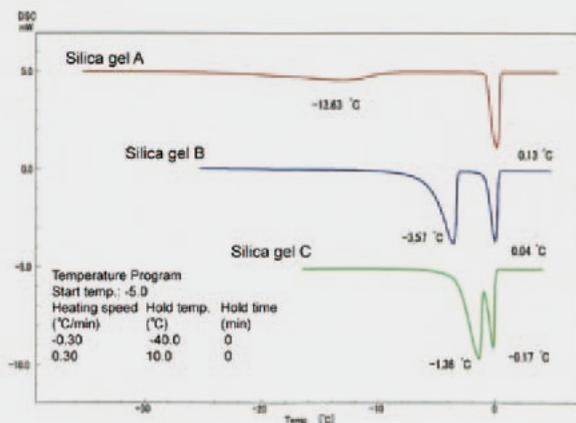
The figure below shows the results from measuring the melting process of water in a solid polymer membrane at various water content levels. At a 6.7% water content, a melting peak for water was not observed. The -23.8°C peak for a water content of 8.5% is presumably due to the melting of water clusters in the solid polymer membrane. The two-stage peak in water content levels at 12.6% and higher is presumably due to peaks for melting free water and water clusters. It shows that as the water content increases, the temperature peak increases. In other words, the amount of free water increases. The temperature and area of the peak for water from clusters melting is probably related to the size of the clusters.



Melting of Water in Polymer Electrolyte Fuel Cell Membrane

Measurement of Water Melting in Silica Gel Pores Using a DSC

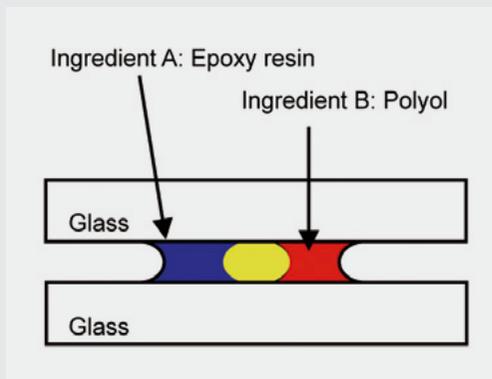
The figure below shows the results from hydrating silica gels A, B, and C with water weighing roughly the same as their respective dry gel weights and then using a differential scanning calorimeter to cool the gels to about -40°C before heating. In each sample, two melting peaks were measured. These presumably correspond to the water in pores melting at a lower temperature and water outside the pores (on the surface) melting at a higher temperature. The peak temperature of pore water melting appears at successively higher temperatures for silica gels A, B, and C. That is probably due to the melting point decreasing as the pore diameter decreases.



Melting of Water in Silica Gel Pores

Near-Infrared Mapping of Adhesives Using an Infrared Microscope

This example uses near-infrared spectroscopy to map a two-solution mixed type epoxy. The epoxy was measured using near-infrared transmission by sandwiching a mixture of appropriate amounts of ingredient A (epoxy resin) and ingredient B (polyol) between two glass slides, as shown below. The image obtained using the mapping software is shown on the right.



Adhesive Sandwiched Between Two Glass Slides

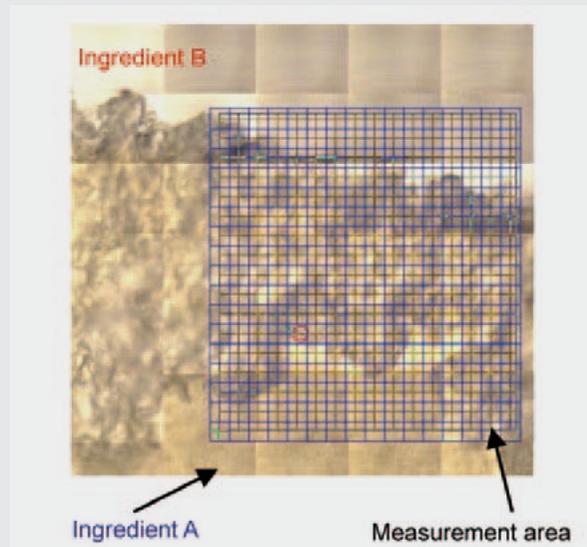
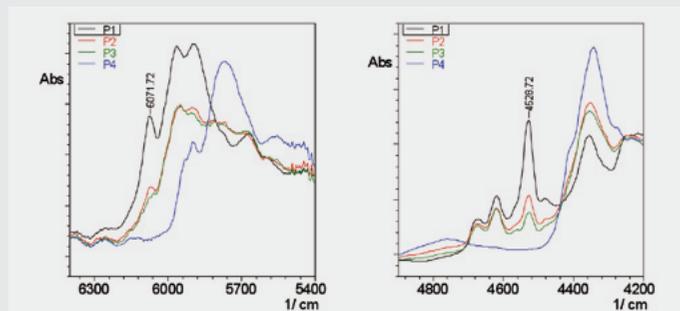
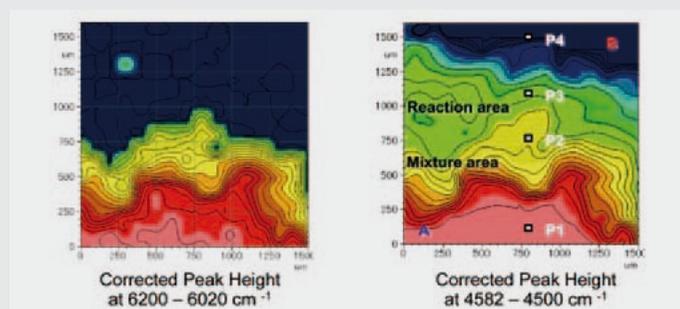


Image After Preview Scanning

Spectra for 4 arbitrary points are shown below, in the upper figure and an intensity map of peak heights at 6071 and 4528 cm^{-1} is shown in the lower figure. The peaks at 6071 cm^{-1} correspond to the first overtone of the C-H stretching vibration on the epoxy group, whereas the peaks at 4528 cm^{-1} correspond to a combination of the C-H stretching and bending vibrations. In the lower intensity map, the peaks at 4528 cm^{-1} are present across the entire A and B mixture area, whereas the peaks at 6071 cm^{-1} are only present near the center of the A and B mixture area. In the area above the center where 6071 cm^{-1} peaks are not observed (side with ingredient B), the epoxy group has presumably reacted with the polyol and changed its structure.



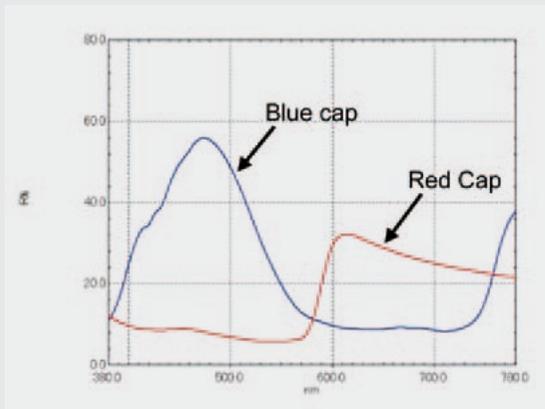
NIR Spectra of 4 Arbitrary Points



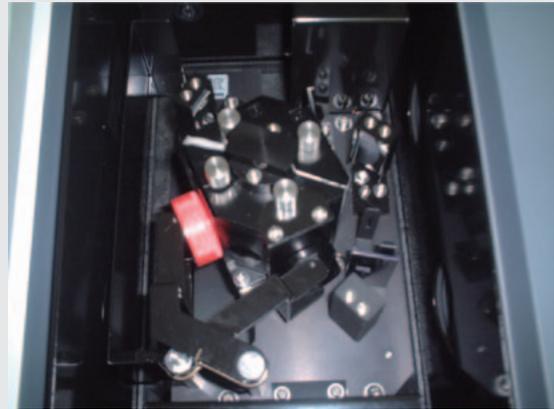
Intensity Map of Peaks at 6071 cm^{-1} and 4528 cm^{-1}

Measurement of Diffuse Reflectance in Plastic Using an Integrating Sphere

In this example, an ISR-2600Plus integrating sphere attachment was installed in a UV-2600 UV-VIS spectrophotometer to measure the diffuse reflectance of two plastic caps (red and blue). The reflectance spectra are shown to the left and a photograph of the sample placed in the integrating sphere is shown to the right. Barium sulfate was used as the reference sample. Based on the left figure, the blue cap mainly reflects blue light in the 400 to 500 nm range, whereas the red cap mainly reflects red light in the 600 to 700 nm range. This system allows measuring the reflectance easily by simply placing the sample in the integrating sphere.



Diffuse Reflectance Spectra of Plastic Caps

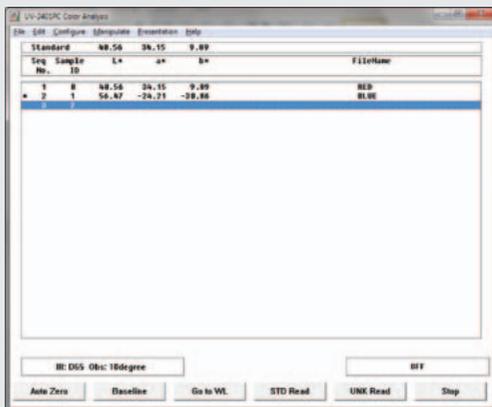


ISR-2600Plus Integrating Sphere

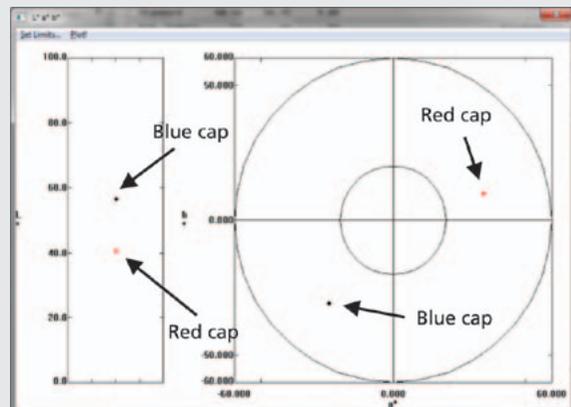
Measuring Color

The color measurement software allows converting spectral data to color values. The figure on the left shows the results from using the color measurement software to convert the above cap data to L*a*b* color system values.

A chromaticity diagram of the L*a*b* color values is shown on the right, where the L* graph shown on the left side indicates the brightness. The higher the data point, the lighter (brighter) the color and the lower the data point, the darker the color. The a*b* graph on the right indicates the color saturation and hue. The closer the data point is to the center of the circle, the less saturated (less colorful) the color and the closer it is to the perimeter, the more saturated the color. Similarly, the radial angle indicates the hue. For example, reds are located toward the upper right. In addition to the L*a*b* color system, the color measurement software can also use various other color systems, such as the Lab, L*u*v*, and Munsell systems.



Main Window of Color Measurement Software

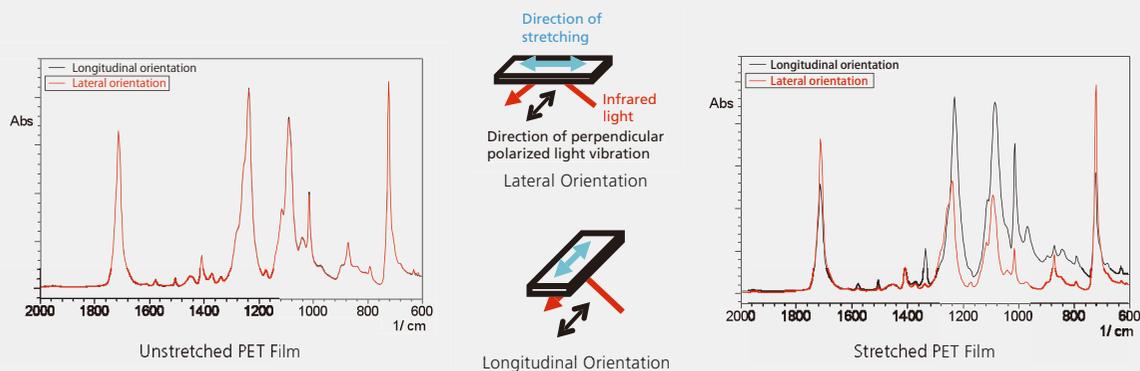


Chromaticity Diagram

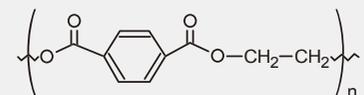
Measurement of Polarized Light of PET Film Using FTIR-ATR Method

When polymers are stretched, the molecules tend to orient longitudinally parallel to the direction they are being stretched. The degree to which they are oriented in the same direction can be determined from polarized light measurements. Film samples are generally measured using the transmission method and polarizers. However, if the film is too thick, the peaks can become saturated, which prevents measurement. In contrast, the ATR method offers the advantage of being able to measure films regardless of their thickness and obtains information about the sample surface as well.

Results from measuring the polarized light of unstretched PET film and PET film stretched to 3 times its original length are shown below. Measurements were performed based on perpendicular polarized light (S), which provides information about vibration perpendicular to the incident plane (the plane formed by the incident and reflected light). Samples were placed in two orientations. One with the direction of stretching parallel to the incident plane (lateral orientation) and the other rotated 90 degrees to a perpendicular position (longitudinal orientation).



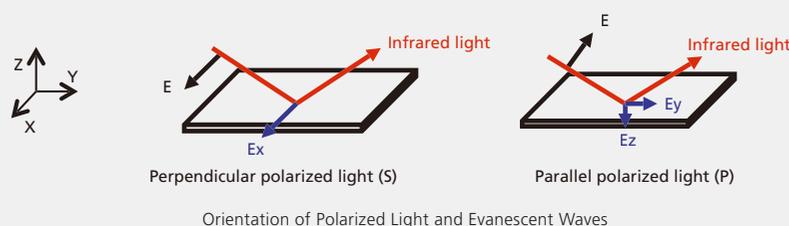
There was almost no difference between the unstretched film spectra and no molecular orientation was observable. However, for the stretched film, there were clear differences in their spectra, such as in the intensity ratio near 1712 cm^{-1} (due to C=O stretching vibration) and near 1248 cm^{-1} (due to C-O stretching vibration). C=O stretching vibration was presumably oriented perpendicular to the molecular axis, whereas the C-O stretching vibration was presumably oriented parallel to the molecular axis. Therefore, these results indicate that the C=O stretching vibration increased when the sample was placed in the lateral orientation, so that the stretching direction was parallel to the incident plane. Similarly, C-O stretching vibration increased when the sample was placed in the longitudinal orientation, so that stretching was perpendicular to the incident plane. Consequently, these results show that the molecules were oriented longitudinally in the stretched film.



Structural Formula for Polyethylene Terephthalate (PET)

Measurement of Polarized Light of PET Film Using ATR Method

Parallel polarized light refers to linear polarized light with an electric field parallel to the incident plane. Conversely, perpendicular polarized light refers to linear polarized light with an electric field in a plane perpendicular to parallel polarized light. For perpendicular polarized light, the evanescent wave generated at the reflection point during ATR measurements has a vector in the X-direction, as shown below. For parallel polarized light, it has vectors in the Y and Z-directions. Therefore, information about molecular vibration in the X-direction can be obtained using measurements with perpendicular polarized light, whereas measurements with parallel polarized light provides information about molecular vibrations in the Y and Z-directions.



Crystallization

Thermal Analysis

Evaluation of Plastic Materials

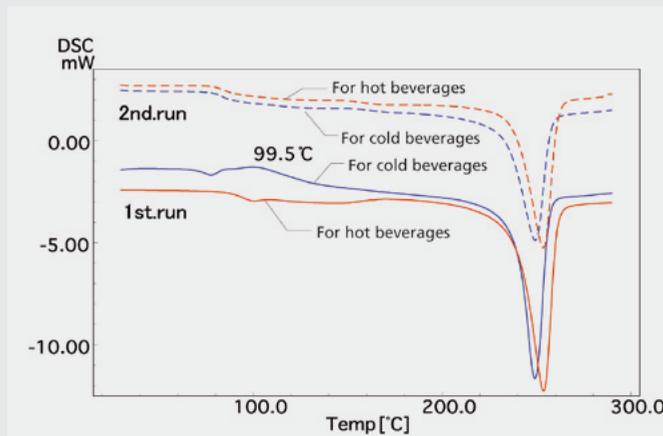
Evaluation of Raw Materials

Product Evaluation

Product Information

Measurement of Commercial PET Bottles

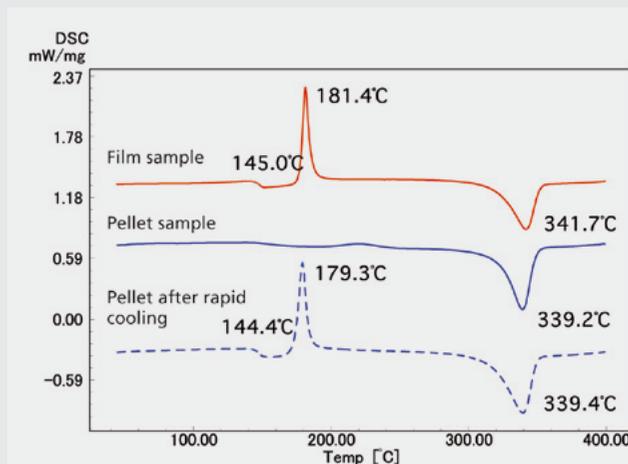
A differential scanning calorimeter (DSC) was used to compare the crystallization in commercial PET bottles for hot and cold beverages. In the first run, an exothermic peak where crystallization presumably occurred, was observed for the cold beverage bottle near 100°C, but was not observed for the hot beverage bottle. In the second run, given the same thermal history, almost no differences were observed between the samples. Based on these results, presumably the hot beverage PET bottle has higher crystallization due to a difference in thermal history experienced during molding. Separate X-ray diffraction analysis results also confirm that the hot beverage bottle has higher crystallization.



DSC Measurement Results of PET Bottles

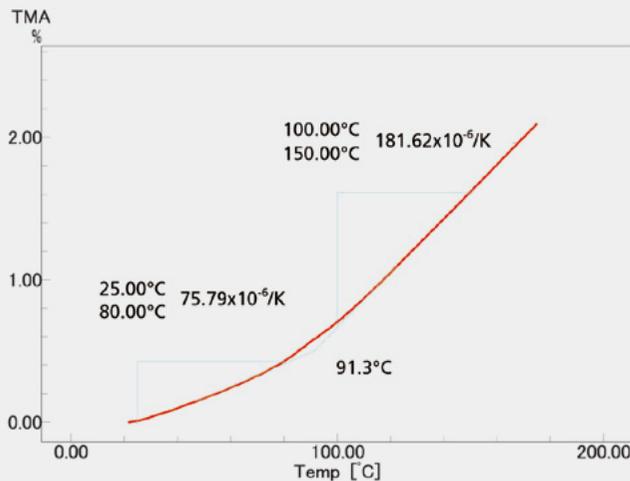
Crystallization of PEEK

This example investigates the relationship between the thermal history of PEEK and its crystallization. The figure below shows that when PEEK film was heated, the glass transition occurred at 145°C, crystallization at 181°C, and melting at 342°C, which indicates it was not crystallized before heating. In contrast, only a melting point of 339°C was measured for the PEEK pellets, which indicates it was already crystallized before heating. This difference in crystallization probably occurred due to the rapid cooling experienced by the film sample during molding, whereas the pellet sample cooled more slowly. This is consistent with DSC measurement results of pellets that were rapidly cooled after the DSC measurement above, which showed the same glass transition, crystallization, and melting temperatures as the film.



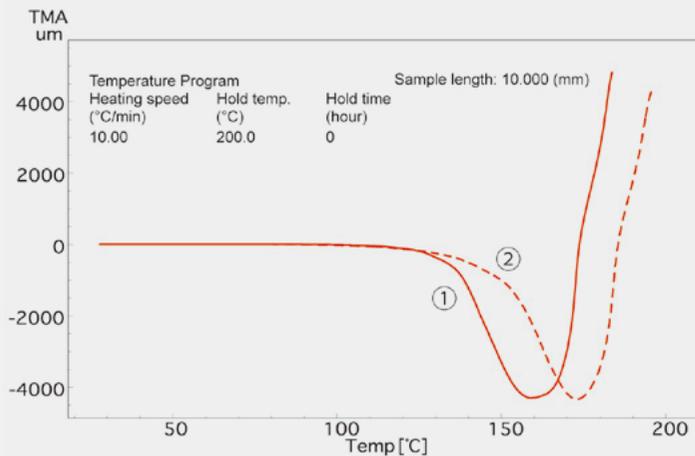
DSC Measurement Results of PEEK

Measurement of Thermal Expansion Process in Epoxy Resin Using a TMA



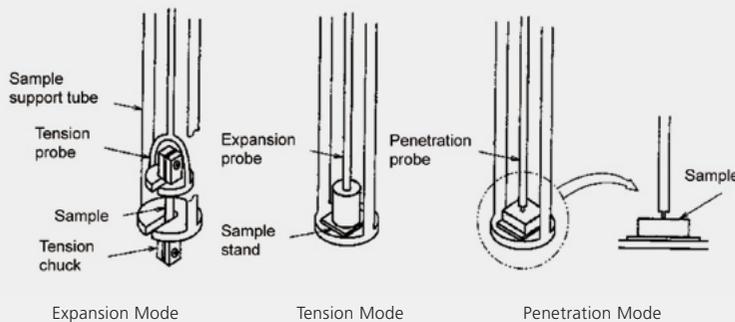
In this example, the thermal expansion process in epoxy resin was measured. The results show a change in the thermal expansion coefficient at about 90°C. This is due to the glass transition of the epoxy resin. Therefore, because material characteristics change at that point, it is necessary to know what that temperature is. Glass transition can also be measured using other methods, such as DSC, but thermomechanical analyzers (TMA) allow measuring the point where mechanical characteristics of the material change.

Measurement of Shrinkage Behavior in PE Film Using a TMA



Two types of PE film were heated under tension. The measurement results indicate that the films began shrinking above 100°C and then began stretching above 150°C. The figure shows that the two types of film shrank at different temperatures. In the tension mode, the strain during shrinking can be measured as well.

Various TMA Measurement Modes



The Shimadzu TMA-60 includes three measurement modes – expansion, tension, and penetration, which can be freely changed depending on the sample shape and objectives. Changing modes is extremely easy, too.

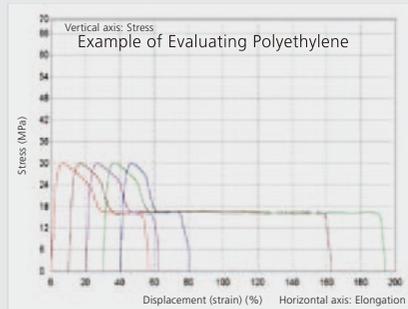
Mechanical Characteristics – Static Strength

Autograph

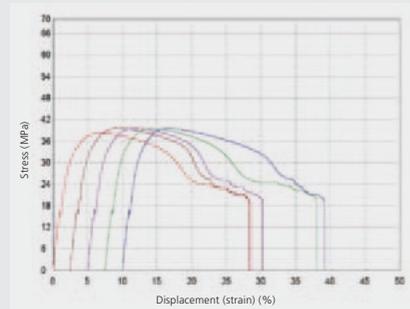
Evaluation of Mechanical Characteristics of Various Plastic Products Using Autograph

The four figures below are examples of results from using a Shimadzu AG-X Autograph precision universal testing machine to evaluate static mechanical characteristics of some typical plastic products. This data allows determining the fundamental characteristic values that are the most important for mechanical design, such as strength, elongation, elasticity, and energy absorption.

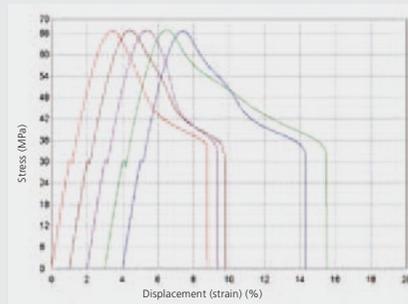
These tests were performed in accordance with JIS standards applicable to the given specimen (sample) shape and test methods.



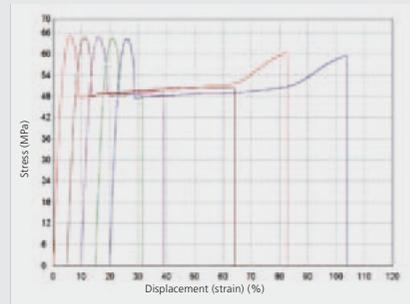
Example of Evaluating Polyethylene



Example of Evaluating Polypropylene



Example of Evaluating Polyvinyl Chloride

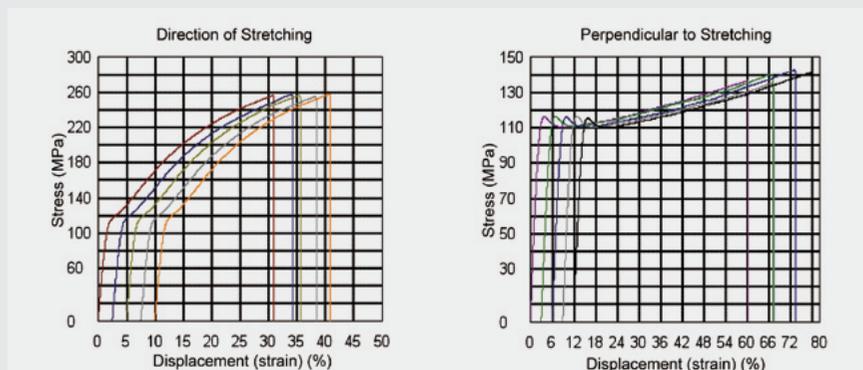


Example of Evaluating Polycarbonate

Evaluation of Plastic Film Characteristics Using Autograph

Thin plastic film materials are increasingly being used for functionally engineered materials. Therefore, it is critically important to understand their characteristics, not only for controlling their functionality and performance, but also for use in developing new applications.

As one example, a 38 μm thick polyester film was evaluated to determine any differences in mechanical characteristics in directions parallel and perpendicular to the direction it was stretched (due to mechanical stretching processes during film manufacturing).



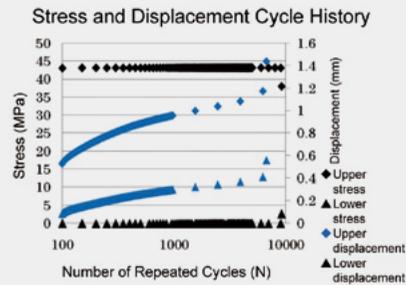
Example of Evaluating Polyester Film

Mechanical Characteristics Fatigue and Impact Strength

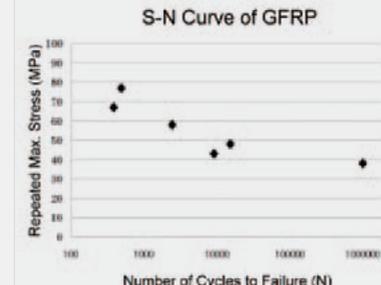
Evaluation of Endurance (Fatigue Strength) of Various Plastic Products Using Servopulser

In actual usage, plastic products are always exposed to some sort of repeated stresses. In particular, if the item is used for transportation equipment or as a structural member, then the endurance must be determined to ensure safety, which involves repeatedly applying a load at high speed to evaluate its service life. Servopulser and Micro-Servo testing machines efficiently and accurately apply loads to evaluate the service life of specimens.

Example of testing endurance of GFRP

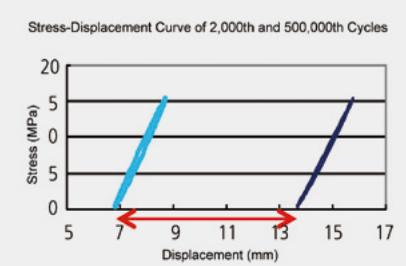


Change in Stress-Displacement Amplitude Over Time

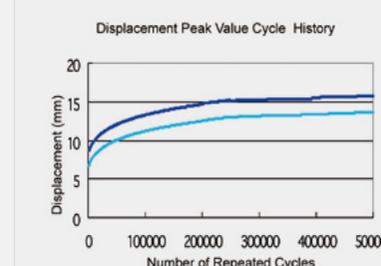


Stress-Endurance Curve

Example of fatigue test of fuel cell electrolyte membrane



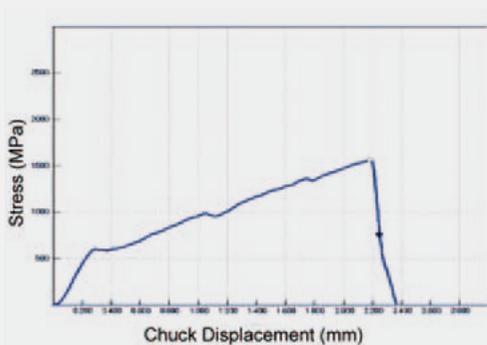
Change in Stress-Displacement Curve Over Time



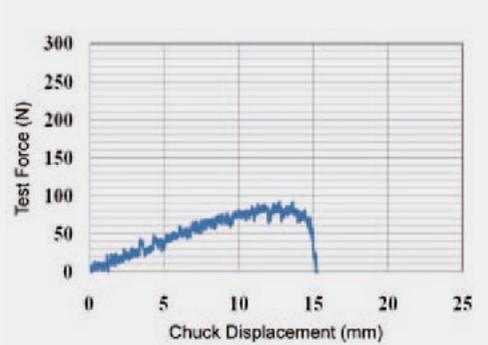
Change in Displacement (Upper/Lower Limits) Over Time

Evaluation of Impact Strength of Various Plastic Products Using Hydroshot

Data for evaluating impact strength is an important parameter for determining the safety and impact (energy) absorbing characteristics of plastic products, such as those used in automobiles. Hydroshot impact testing machines obtain test force and displacement data at loading speeds of up to 20 m/sec (72 km/h), where loading can be applied in either tensile or puncture modes.



High-Speed Tensile Testing of CFRP



High-Speed Tensile Testing of Polyester Film

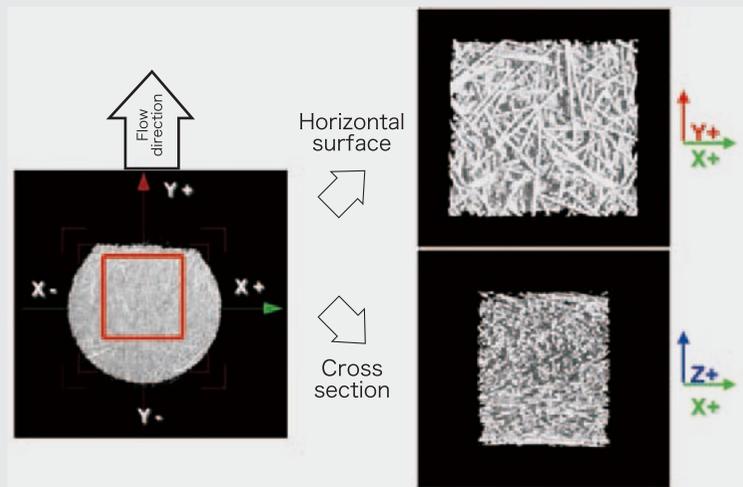
Non-Destructive Inspection Defect Inspection and Internal Structural Analysis

Example of Analyzing and Observing the Internal Structure of Various Plastic Products Using Industrial X-Ray Inspection

- Analyzing the interior of glass fiber reinforced plastic

The following is an example of analyzing the interior of glass fiber reinforced plastic (GFRP).

Using a CT image to observe the fiber orientation inside the material showed that the fibers were oriented differently in the surface and center layers. Fibers in the surface layer were oriented parallel to the flow direction (direction the plastic material flowed during manufacturing), whereas fibers in the center layer were oriented perpendicular to the flow. This result is also consistent with computer analysis. Consequently, information about fiber orientation that was previously obtained by making FRP specimens and inspecting them visually (or with a microscope) can now be obtained non-destructively.

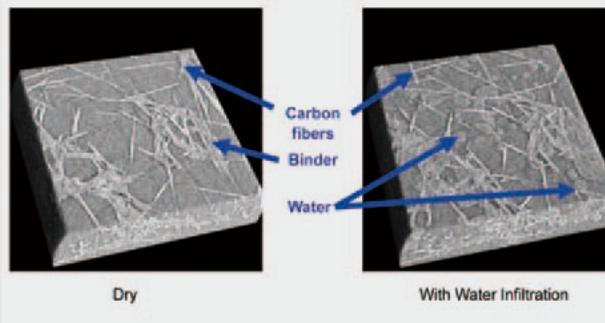


Example of Observing Internal Structure of GFRP (Fiber Orientation)

Example of Analyzing and Observing the Internal Structure of Various Plastic Products Using Industrial X-Ray Inspection

- Observing water inside a fuel cell

The following is an example of observing water in the gas diffusion layer of a polymer electrolyte fuel cell. This shows that the presence of water can clearly be determined and the formation, distribution, and movement of water can be observed non-destructively. Therefore, this inspection is expected to make a significant contribution to the development of fuel cells.



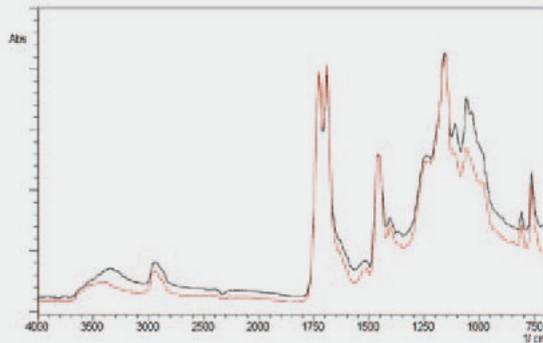
Example of Observing Internal Structure of Fuel Cells (Distribution of Water)

Analysis of Foreign Substances on Film Using an Infrared Microscope

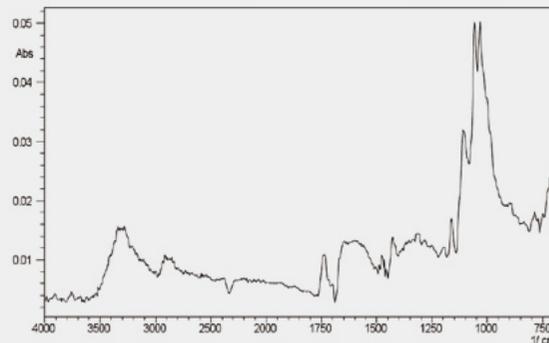
The following are results from using an infrared microscope and the attenuated total reflection (ATR) method to analyze trace contaminants discovered on the surface of film. The fibrous substance, which was about 5 μm wide, and the film surface were both measured to obtain a differential spectrum. From the resulting spectrum it was determined that the fibrous substance was cellulose fibers. In this way, an infrared microscope and ATR method can be used to obtain information about sample surfaces, such as difficult-to-sample substances adhered to sample surfaces, without any pretreatment.



Microscope Photograph of Foreign Substance on Film
Red box: 50 \times 100 μm



Measurement Results from Foreign Substance (Black) and Normal Area



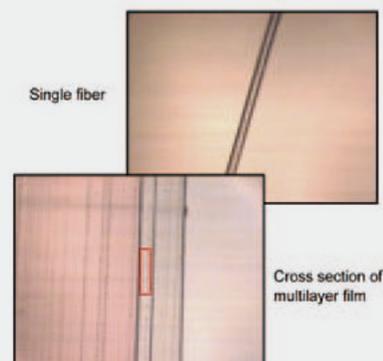
Differential Spectrum (Cellulose)

Infrared Microscope

Infrared microscopes focus condensed infrared light on a sample and detect the light passed through or reflected from the sample with a highly sensitive MCT detector. This enables measuring very small samples and micro areas that cannot be analyzed using the FTIR system alone. Transmission, specular reflectance, ATR or other methods can be selected depending on the measurement sample and purpose of analysis, which is extremely helpful for analyzing defects, such as foreign substances or deterioration, identifying the respective layers of multilayer films, or analyzing micro-areas. Samples can be measured (transmittance or specular reflectance measurement) by viewing the sample in the same manner as operating an optical microscope, and then specifying the measurement location using the auto aperture. Unlike other instruments that analyze micro-areas by irradiating samples with high-energy visible near-infrared laser light, X-rays, or electrons, infrared microscopes cause almost no change or damage to samples.



Infrared Microscope System



Photographs from Infrared Microscope

Analysis of Cadmium in Plastics Using an AA Spectrophotometer

Atomic absorption (AA) spectrophotometers are able to analyze elements in solution with high sensitivity. Analysis of solid samples, such as plastics, requires preparing a sample solution using pretreatment methods such as dry incineration or Kjeldahl digestion (wet digestion). In this case, standard polyethylene samples BCR680 and 681 were prepared by dry incineration and Kjeldahl digestion and results were also compared to those obtained by ICP emission spectrometry.

(1) Dry Incineration Method

Weigh 0.2 g of sample into a quartz crucible. Add enough sulfuric acid to immerse the sample. Heat the crucible on a hotplate until the white SO_3 smoke starts dissipating and then place it in an electric furnace and incinerate the contents to ash at 450°C . After incineration, add 5 mL of hydrochloric acid (1 + 2) to the residue and evaporate it to dryness on a water bath. Add 10 mL of 1 mol/L nitric acid and apply heat to dissolve the sample. After letting it cool, measure 20 mL of the digest solution. (Reference: Food Sanitation Inspection Guidelines – Physics and Chemistry, 2005, supervised by the Japanese Ministry of Health, Labour, and Welfare)

(2) Wet Digestion Method

Weigh 0.2 g of the sample in a Kjeldahl flask. Add sulfuric acid, nitric acid, and hydrogen peroxide to the flask and heat it on a heating mantle (at about 300°C) to digest the sample. The contents will become black with carbon and white SO_3 smoke is emitted. After the solution turns black with carbon, add more nitric acid and hydrogen peroxide and continue heating (at about 350°C). Repeat this process until the contents turn pale yellow. After letting the flask cool, measure 20 mL of the solution. (Reference: BS EN1122 Method A: 2001)

Analytical Conditions and Results

Measurement Results

Measurement results were obtained using the flame atomization method. The cadmium (Cd) calibration curve used for measurements is shown in Fig. 1. Table 1 shows a comparison of data obtained using different pretreatment and measurement methods (ICP emission spectrometry). These show that both methods gave results that were roughly consistent with certified values. Given flame atomic absorption spectrophotometry is used and the sample is diluted by 100 times before measuring, the lower quantitation limit in plastic is 0.5 mg/kg.

Analytical Conditions

Analytical wavelength: 228.8 nm
Slit width: 0.7 nm
Measurement mode: BGC-D2
Acetylene gas flowrate: 1.8 L/min
Air flowrate: 15 L/min

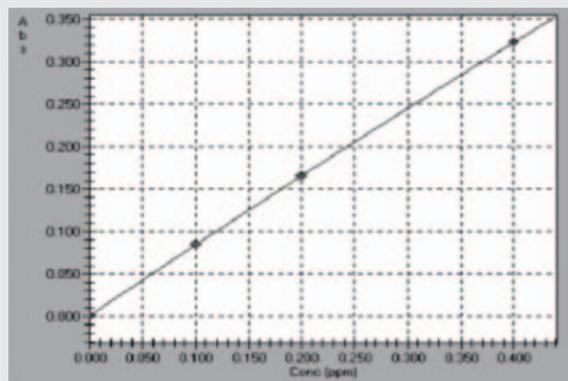


Fig. 1 Calibration Curve for Cd by Atomic Absorption Spectrophotometry

Table 1 Quantitation Results of Polyethylene

	Dry incineration		Wet digestion		Certified value (mg/kg)
	AA	ICP	AA	ICP	
BCR680	140	141	142	140	140.8
BCR681	20.0	21.0	22.0	21.4	21.7

Analysis of Hazardous Metals in Plastics Using an ICP-AES System

ICP emission spectrometers are able to analyze elements in sample solutions with high sensitivity. Solid samples, such as plastics, require using dry incineration, wet digestion, microwave digestion, or other methods to pretreat samples to prepare a solution. However, that requires selecting the appropriate pretreatment method for the elements being analyzed. This example introduces results from using respective pretreatment methods to prepare solutions of plastic samples (standard polyethylene samples BCR680 and BCR681) and then analyze them for hazardous metals, such as lead (Pb) and cadmium (Cd), using a Shimadzu multitype ICPE-9000 emission spectrometer.

Sample Pretreatment Methods

(1) Dry Incineration Method

Weigh 0.2 g of sample into a quartz crucible. Add enough sulfuric acid to immerse the sample. Heat the crucible on a hotplate until the white SO₃ smoke starts dissipating and then place it in an electric furnace and incinerate the contents to ash at 450°C. After incineration, add 5 mL of hydrochloric acid (1 + 2) to the residue and evaporate it to dryness on a water bath. Add 10 mL of 1 mol/L nitric acid and apply heat to dissolve the sample. After letting it cool, measure 20 mL of the digest solution. (Reference: Food Sanitation Inspection Guidelines – Physics and Chemistry, 2005, supervised by the Japanese Ministry of Health, Labour, and Welfare)

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(3) Microwave Digestion Method

Weigh 0.2 g of the sample in a digestion vessel. Add nitric acid and hydrogen peroxide and seal the vessel. Digest the sample in microwave sample pretreatment unit. After letting the digestion vessel cool, measure 20 mL of the digest solution. Note: For samples containing large amounts of coexisting components, such as additives, use a small amount of hydrofluoric acid. (Reference: US EPA SW-846 Method 3052)

Analytical Conditions and Results

Analytical Results

Analytical results are shown in Table 1. Good results were obtained, with lead (Pb) and cadmium (Cd) results from dry incineration, cadmium (Cd), total chromium (Cr), and mercury (Hg) results from wet digestion, and results for all elements from microwave digestion consistent with certified values.

Discussion

The ICP emission spectrometer enabled measuring trace components with good precision. Nevertheless, Hg results were low for dry incineration, presumably due to scattering at high temperature and Pb results were low for wet digestion, presumably due to precipitation of lead sulfate formed from reaction with the sulfuric acid used for digestion. Therefore, an appropriate sample pretreatment method must be selected based on the elements being measured.

Analytical Conditions

Instrument: ICPE-9000
 High frequency output: 1.2 kW
 Plasma gas flowrate: 10 L/min
 Auxiliary gas flowrate: 0.6 L/min
 Carrier gas flowrate: 0.7 L/min
 Sample introduction: Coaxial nebulizer
 Spray chamber: Cyclone chamber
 Plasma torch: Mini torch
 Observation method: Axial

Table 1 Quantitation Results of Polyethylene

Unit:mg/kg

sample Pretreatment Element	BCR680				BCR681			
	Dry incineration	Wet digestion	Microwave digestion	Certified value	Dry incineration	Wet digestion	Microwave digestion	Certified value
Cd	141	140	140	140.8	21.0	21.4	21.7	21.7
Pb	105	< 0.2	108	107.6	13.1	< 0.2	13.5	13.8
Cr	105	112	112	114.6	16.2	17.2	17.5	17.7
Hg	< 0.2	24.0	25.6	25.3	< 0.2	4.3	4.6	4.5
As	28	31	30	30.9	4	4	4	3.93

Hazardous Substances Hazardous Metals

UV

Evaluation of Plastic Materials

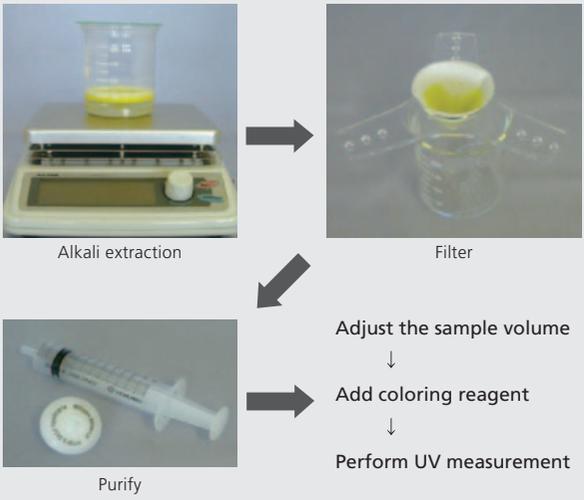
Evaluation of Raw Materials

Product Evaluation

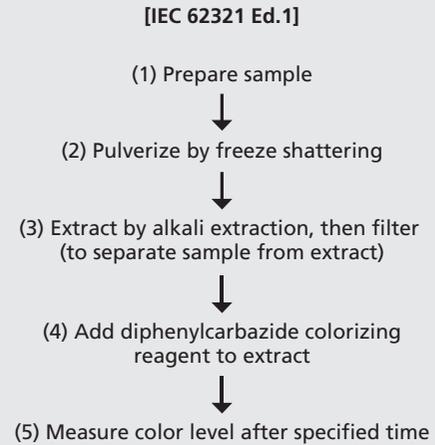
Product Information

Analysis of Hexavalent Chromium in Plastic Using a UV-VIS Spectrophotometer

The content of hexavalent chromium is regulated by the ELV, WEEE, and RoHS directives, which regulate hazardous substances in Europe (EU). Hexavalent chromium in plastic is analyzed by pretreating the sample to extract the hexavalent chromium into solution and then analyzing the solution by colorimetry using a UV-VIS spectrophotometer. A process flowchart for extracting the hexavalent chromium from plastic is shown to the right.



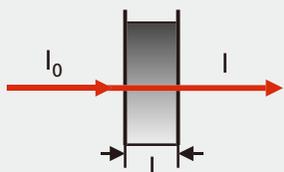
Process Flow of Actual Analysis



Process Flowchart of Extracting Hexavalent Chromium from Plastic

Colorimetric Analysis Using a UV-VIS Spectrophotometer

UV-VIS spectrophotometers disperse light from a light source with a grating to irradiate samples with the light, and then measure how much light the sample absorbed based on the difference in light energy level before and after irradiating the sample. Hexavalent chromium is analyzed by extraction into a solution and then colorization using diphenylcarbazide reagent. After colorization of the hexavalent chromium, the spectrophotometer measures the level of color.



Beer-Lambert Law

$$\text{Transmittance } T = I/I_0 = 10^{-\epsilon c l}$$

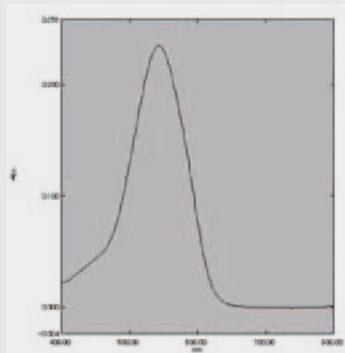
- ϵ : Absorption coefficient
- c : Solution concentration
- l : Optical path length

$$\text{Absorbance } A = \log(1/T) = \epsilon c l$$

The solution concentration is proportional to the absorbance.



Diphenylcarbazide



Absorption Curve of Cr(VI) Diphenylcarbazide Complex

UVmini-1240 Hexavalent Chrome Analysis



Hazardous Substances Organic Substances

Analysis of Phthalate Esters in Plastic

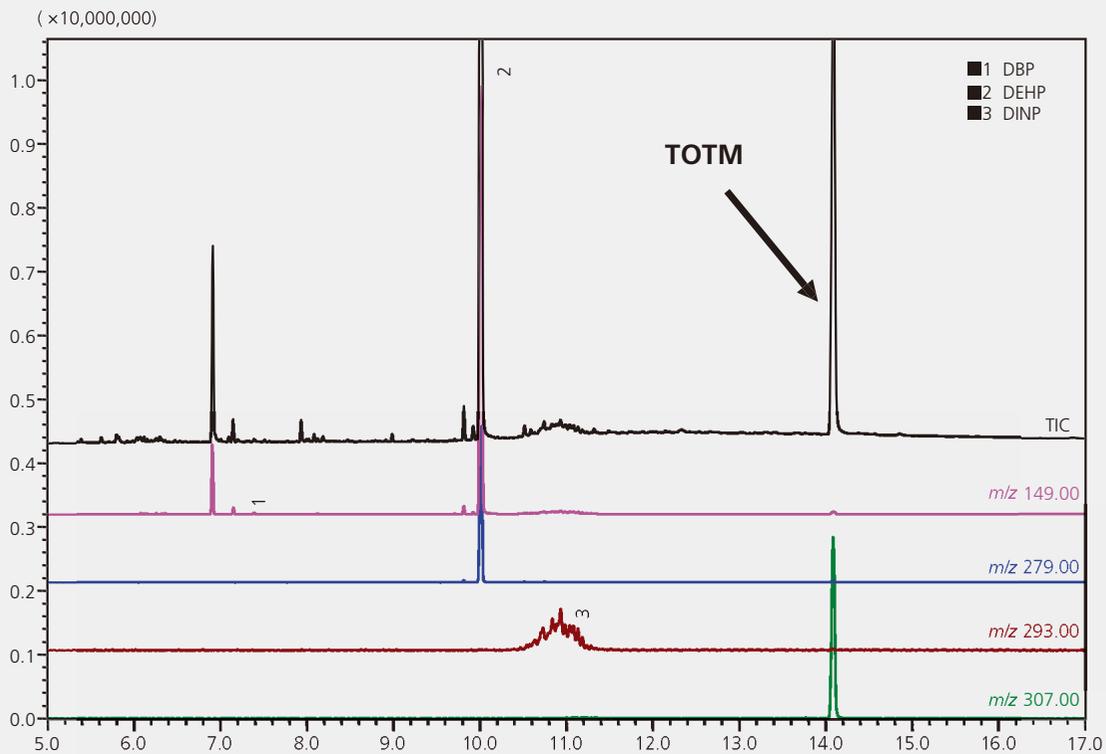
Various countries have established regulations to restrict the use of phthalate esters, which are commonly added as plasticizers in plastic toys and consumer goods. Some of the phthalate esters subject to regulation include DEHP (bis(2-ethylhexyl) phthalate), DBP (dibutyl phthalate), BBP (butyl benzyl phthalate), DINP (di-isononyl phthalate), DIDP (di-isodecyl phthalate), and DNOP (di-n-octyl phthalate). The analytical method typically involves extracting the phthalate esters from the plastic using solvents and analyzing the extract "as is" or diluted. If concentration levels are relatively high and there are few interfering substances, then GC-FID can be used for analysis, but plastic samples containing large numbers of interfering substances or phthalate esters such as DINP or DIDP, which have many isomers, are analyzed using GC/MS.

Instrument Configuration

GCMS-QP2010 Ultra
AOC-20i/s autoinjector

Analytical Conditions

The sample (about 1 g of plastic) is immersed in 50 mL of acetone/hexane (3:7) at 40°C and left over night. Then the extract solution is diluted to a known volume and injected into the GC/MS system.



Analysis of Hazardous Elements

EDX

Evaluation of Plastic Materials

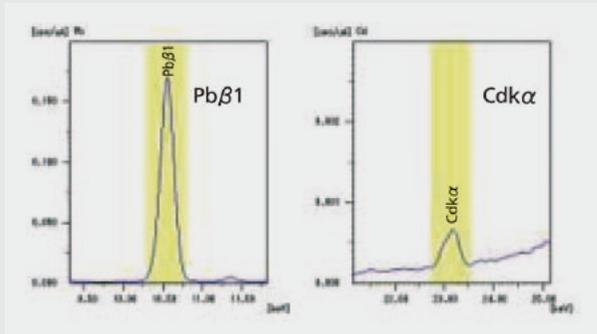
Evaluation of Raw Materials

Product Evaluation

Product Information

Analysis of Hazardous Elements in Plastic Using an EDX Spectrometer

Due to its ability to quickly, easily, and non-destructively analyze solids, powders, and liquids, X-ray fluorescence spectrometers have been widely used for screening the five elements (Cd, Pb, Hg, Cr, and Br) regulated under the RoHS and ELV directives. This example measures hazardous elements in the insulating jacket material of a LAN cable. Specifically, it shows results from analyzing Pb and Cd.



EDX-720 Spectra for Pb (Left) and Cd (Right)



LAN Cable

Quantitation Results Using an EDX System

Pb: 7600 ppm

Cd: 52 ppm

Analytical Conditions

Instrument: EDX-720

Tube voltage: 50 kV

Tube current: Automatic

Measurement time: 100 sec

Optimal filter used

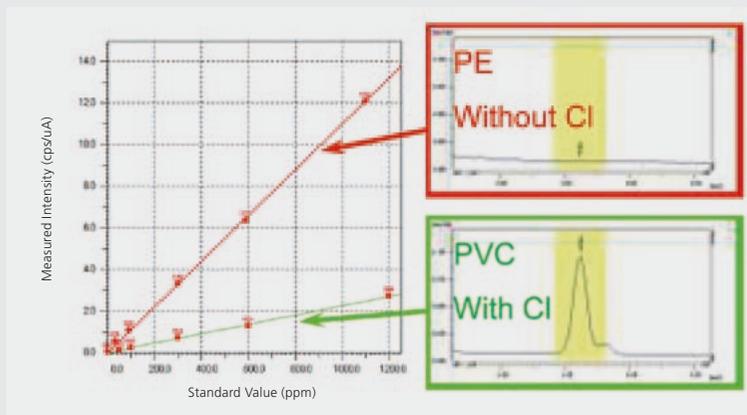
A calibration curve was created and quantitative analysis performed using standard samples prepared by adding the five RoHS elements (Cd, Pb, Hg, Cr, and Br) to PE and PVC plastic.

Features of the EDX Series

EDX series models are equipped standard with a primary filter that is optimal for hazardous elements, which allows measuring the five RoHS elements easily and with high sensitivity. In addition, a background internal standard correction function is included to correct for the thickness, shape, and size of samples. Background internal standard correction corrects quantitation values based on the ratio between continuous X-rays from the X-ray tube or characteristic X-rays from the target and scattered rays.

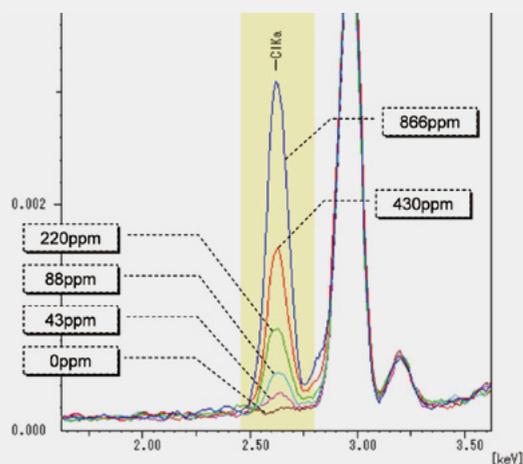
Automatic Calibration Curve Selection Function

When analyzing X-ray fluorescence, different sample types (PE versus PVC, for example) cause the X-ray fluorescence intensity to vary. The automatic calibration curve selection function allows using the software to determine the presence of Cl, automatically select the optimal calibration curve for PE or PVC, and then quantitate the content.

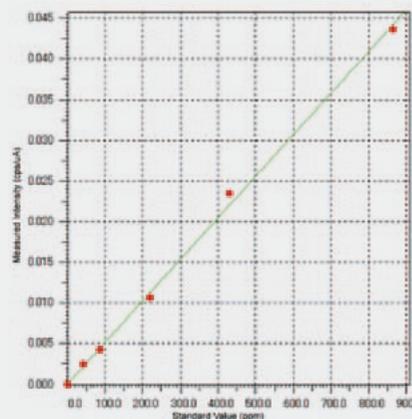


Analysis of Halogen Elements (Chlorine) Using an EDX Spectrometer

Due to its ability to analyze solids, powders, liquids, and other samples quickly, easily, and non-destructively, X-ray fluorescence analysis has been widely used for screening elements regulated by RoHS and ELV directives. More recently, the electrical/electronic industries have been promoting the elimination of halogen elements, such as BR and Cl, from their products, in addition to the five elements subject to the RoHS directive. In this example, Cl was analyzed in plastic.



Cl Spectra



Calibration Curve for Cl Kα

The calibration curve was created using a standard sample prepared by adding Cl to polyethylene (PE) plastic.

Analytical Conditions

Instrument: EDX-720
 Spectrum: Cl Kα
 Tube voltage: 15 kV
 Tube current: Automatic
 Measurement time: 100 sec
 Optimal filter used

Features of the EDX Series

The primary filter included standard for Cl allows easily measuring Cl with high sensitivity. Also, attaching a vacuum measurement unit (option) allows analyzing Cl with even higher sensitivity.

Analysis

The Cl content in a plastic plate specimen and plastic parts was measured. In addition, shape correction based on the background internal standard correction function was used to correct for the thickness, shape, and size of samples. Background internal standard correction corrects quantitation values based on the ratio between continuous X-rays from the X-ray tube or characteristic X-rays from the target and scattered rays.

	Plastic plate	Part (white)	Part (yellow)
Sample			
Cl quant. value	86.7	191	239

(Unit: ppm)

Fourier Transform Infrared Spectrophotometer

Analysis of Plastic Samples by FTIR

Many different types of information can be obtained from plastic samples using FTIR systems, such as information for checking raw materials, the composition of plastic pellets or powders, bonding modes, isotope ratios, copolymer composition ratios, molecular orientation, and information for analyzing foreign matter or other defects. Since FTIR allows analyzing samples easily and quickly, and also allows analyzing micro areas and surfaces, it is especially useful for analysis and testing in various plastic product manufacturing processes.

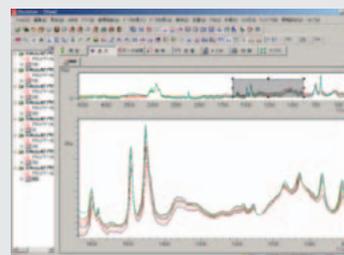
Features of the Shimadzu IRAffinity-1 Fourier Transform Infrared Spectrophotometer

- Higher sensitivity and performance than general purpose systems
- IRsolution software is easy to operate and offers sophisticated functionality
- Expanding range of applications

This attractive and compact Fourier transform infrared spectrophotometer features a dynamic alignment mechanism that keeps the interferometer continuously optimized and a built-in dehumidifier that ensures easy maintenance. It offers an S/N ratio of 30,000:1 (given 4 cm^{-1} resolution, 1 minute accumulation, near 2100 cm^{-1} , and peak-to-peak), maximum resolution of 0.5 cm^{-1} , and a compact design. In addition, the IRsolution high-functionality, high-operability software and analysis support programs (Contaminant Analysis Program and Pharma Report Program) ensure data processing and postrun analysis can be accomplished easily.



IRAffinity-1



IRsolution High-Functionality Software

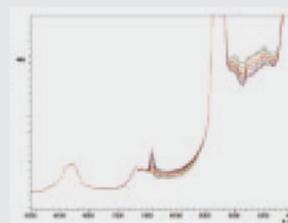
Features of the Shimadzu IRPrestige-21 Fourier Transform Infrared Spectrophotometer

- High sensitivity and high functionality
- High reliability
- Measurement range from near infrared to far infrared
- IRsolution high-functionality, high-operability software

High-throughput optics, with features such as a high-energy ceramic light source, temperature-controllable high-sensitivity DLATGS detector, and gold-coated mirrors, achieves high S/N ratios above 40,000:1 (given a 4 cm^{-1} resolution, 1 minute accumulation, near 2100 cm^{-1} , and peak-to-peak). In addition, an advanced dynamic alignment (ADA) mechanism keeps the interferometer continuously optimized and a built-in dehumidifier ensures easy maintenance. A wide measurement range, from near infrared to far infrared, and a wide assortment of accessories expand the possible range of applications. In addition, the IRsolution high-functionality, high-operability software and analysis support programs (Contaminant Analysis Program and Pharma Report Program) ensure data processing and postrun analysis can be accomplished easily.



IRPrestige-21

Near Infrared Spectrum of Octanoic Acid
(tracking changes in hydrogen bonds due to heat)

Single Nano Particle Size Analyzer

Product Information

Evaluation of Raw Materials

Analysis of Plastic Samples by IG-1000 Single Nano Particle Size Analyzer

Biological macromolecules, nanoparticles, and macromolecular nanoparticles made by combining such components are actively being developed in drug formulation and other fields. For such nano-sized particles, the particle size distribution is one of the most important physical properties. The IG-1000 Single Nano Particle Analyzer utilizes the induced grating (IG) technology developed independently by Shimadzu for measuring the distribution of single-digit nanoparticles dispersed in liquid with high sensitivity and good reproducibility.

Features of the IG-1000 Single Nano Particle Size Analyzer Measurement Range: 0.5 nm to 200 nm

- Measures the distribution of single-digit size nanoparticles dispersed in liquid with high sensitivity and reproducibility.
- Accurately measures samples with broad size distributions.
- Free from the tendency to overstate presence of large particles and not be able to measure small particles.
- Measurement results are unaffected by trace amounts of large particles outside the measurement range (such as aggregates or contaminants).
- Samples can be measured in normal environments, even without any careful pretreatment (filtering) processes.
- Allows using raw data (time course variations in intensity of diffracted light) to verify the validity of measurement results.

Particle diameters are determined by forming a diffraction grating from the particles and then measuring diffusion speed.

The particle diffraction grating is formed using dielectrophoresis.

Samples are placed in a batch cell, then a comb-shaped electrode substrate is inserted into the cell. When an alternating current is applied to the comb-shaped electrodes, dielectrophoresis concentrates the particles dispersed throughout the liquid into a particle diffraction grating pattern. When the alternating current is switched OFF, the dielectrophoresis stops and the particle diffraction grating pattern begins diffusing.

Diffusion speed is determined from the time course change in primary diffracted light intensity.

Primary diffracted light is detected by irradiating the particle diffraction grating with laser light. The primary diffracted light intensity decreases correspondingly as the particle diffraction grating diffuses. In this way, the diffusion speed of the particle diffraction grating is monitored based on the change in primary diffracted light intensity as a function of time.

In previous systems based on light scattering methods, which use light scattered from particles as the detected signal, the signal level drops rapidly as the particle size decreases. That makes it extremely difficult to measure single-digit nanoparticles. In contrast, the IG method uses the primary diffracted light from the macro structure of a particle diffraction grating as the detection signal. Therefore, the signal level is not dependent on particle size, making it possible to obtain more than adequate signal even from single-digit nanoparticles, which in turn makes it possible to measure particle size distributions with high sensitivity and reproducibility.

Particle size is determined from the relationship between particle size and diffusion speed.

Large particles diffuse slowly and small particles, particularly single-digit nanoparticles, diffuse quickly. The IG method determines particle size from the relationship between particle size and diffusion speed.



IG-1000 Single Nano Particle Size Analyzer

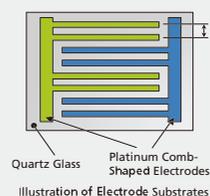
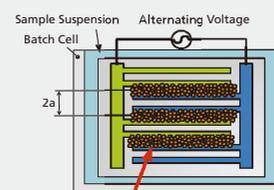
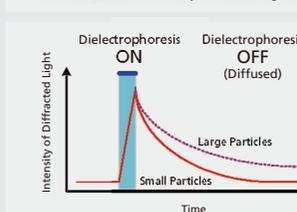
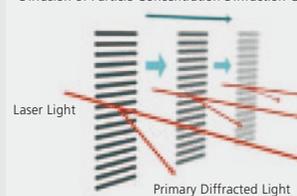


Illustration of Electrode Substrates



Diffraction Grating Formed from Particles (pitch is twice the pitch of the comb-shaped electrodes)

Diffusion of Particle Concentration Diffraction Grating



Evaluation of Plastic Materials

Evaluation of Raw Materials

Product Evaluation

Product Information

High-Performance Liquid Chromatograph

Analysis and Fractionation of Additives and Impurities Using HPLC and Preparative LC Systems

Additives to polymer materials and products have various important functions that can determine product performance, such as heat resistance or structural integrity. Impurities can affect the quality of products, making them less stable or resilient. HPLC systems are used to separate and quantitate many different additives and impurities, whereas preparative LC systems are used to separate and fractionate large quantities of additives and impurities for purposes such as structural analysis.

Features of Shimadzu Prominence Series High-Performance Liquid Chromatographs

The Prominence series was developed to improve the efficiency of analytical processes and improve operating and data reliability. Prominence HPLC systems offer new features and outstanding performance compared to previous models, such as Web-based monitoring and control, high-speed sample injection, and high-sensitivity detection.

- Improved Analytical Productivity
 - Fastest sample injection (10 sec for 10 μ L)
 - Fully automated startup and shutdown
 - **Self-diagnostic and self-repairing functions (expert function)**
 - Energy saving auto-validation function
 - Web-control (allows access to the instrument via an Ethernet connection)

- Improved Analytical Reliability
 - High sensitivity and extended linearity
 - Nearly zero cross-contamination
 - Improved detector stability with a thermostatic flow cell
 - Improved flowrate accuracy



Prominence Series

Features of Shimadzu Prominence Semi-Preparative Recycling High Performance Liquid Chromatographs

- The **semi-preparative recycling system** includes a kit that minimizes the internal volume, which results in highly efficient separation by closed loop recycling.
- By combining this system with appropriate columns, it is possible to achieve more than several million theoretical plates.



Prominence Semi-Preparative Recycling System

Analysis of Plastic Samples by PY-GC/MS

Pyrolysis gas chromatography mass spectrometry is an especially powerful analytical technique for researching new materials, polymer characterization, and other applications. That is because pyrolysis gas chromatography enables analyzing polymer compounds—such as in insoluble or composite materials, and in ultra trace quantities—without requiring any complicated pretreatment. In addition, it provides unique information that is difficult to obtain using other methods.

Features of the GCMS-QP2010 Ultra

The GCMS-QP2010 Ultra features a high-efficiency ion source, detector with overdrive lens, and high-capacity differential vacuum mechanism to provide a system with high sensitivity and high stability. The highly sensitive and exceptionally stable GC/MS system decreases the lower detection and quantitation limits, which increases the hit rate for mass spectra similarity searches for even ultra trace components. Furthermore, decreasing the absolute sample quantity injected allows simplifying concentration pretreatment and reducing contamination in injection port inserts and columns. The newly designed electronic control platform offers the fastest scan speeds in class. That means it is also perfect for other applications that require fast scan speeds, such as simultaneous scan/SIM measurements or GC × GC/MS and fast-GC/MS analysis.



GCMS-QP2010 Ultra

Features of the Pyrolysis System (for polymer compound analysis)

Pyrolysis-GC/MS is commonly used to measure samples related to plastics. It performs analysis on plastics, rubbers, and other polymer compounds by decomposing them at temperatures above 500°C and using GC/MS to analyze the resulting thermal decomposition products. Since the decomposition products reflect the structure of the original polymer compound, they can be used to identify the polymer or perform more sophisticated structural analysis. An F-Search feature (polymer and additive search software) is also provided to help identification. In addition to the features of a conventional pyrolysis system, the double-shot pyrolyzer also enables selecting more sophisticated analytical methods, such as evolved gas analysis with programmed heating or thermal desorption with flash pyrolysis.



GCMS-2010 Ultra
EGA/PY-3030D

System Contents

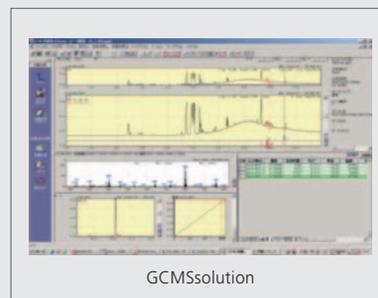
GCMS-QP2010 Ultra, EGA/PY-3030D, AS-1020 sampler, etc.

Applications

Plastics, rubbers, polymers, and other macromolecule compounds, and trace organic impurities in inorganic materials

Features of GCMSsolution

The GC/MS workstation software, GCMSsolution, offers extensive functionality combined with ease of use. Intuitive controls, assistant bar and data explorer features for providing data management functions, Word-like report functions, and in-depth GLP/GMP support functions all contribute to a software package that helps improve analytical productivity.



GCMSsolution

Liquid Chromatograph Mass Spectrometer

Analysis of Polymer Additives Using LCMS

The various types of additives added to polymer materials and products serve an important function that can determine that product's performance. For example, for the same type of polymer, the formulation of additives can vary depending on the brand or grade. This means that the information obtained from analyzing additives can be very important for market research or product development.

Features of Shimadzu LCMS-2020 Liquid Chromatograph Mass Spectrometers

From HPLC to UFLC

In addition to speed and separation capabilities, ultra fast LC (UFLC) succeeds in offering both higher precision and expandability than possible with conventional high-performance LC (HPLC) systems.

Ultra Fast Speed

To achieve overall faster processing speeds, the LCMS-2020 offers not only faster analysis, but also faster sample injection speeds and fully automated analytical functions.

Exceptional Reproducibility

Even though the LCMS-2020 is ultra fast LC, it provides very high injection reproducibility. It also offers unrivaled carryover suppression, which is critical for LC/MS analysis.

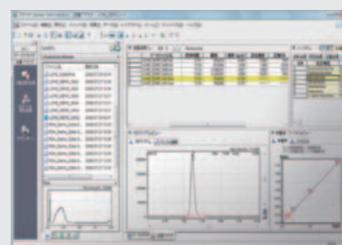
Extensive Expandability

In addition to ultra-fast analysis, the system also offers the flexibility to be used for many other applications as well, such as normal HPLC or semi-preparative LC.

Using these features allows easily confirming the elution of components based on the *m/z* value of known additives, highly selectively quantitating components, predicting the molecular weights of unknown compounds, and more.



LCMS-2020



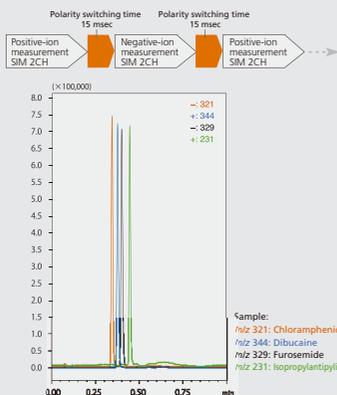
LCMSsolution Ver. 5 for LCMS-2020

Ultra-fast UFs switching

Rapid 15-millisecond positive/negative ionization switching

To detect both positive and negative ions, analysis is performed while switching between the positive and negative ionization modes.

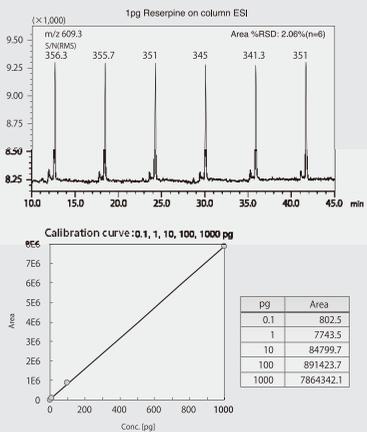
The LCMS-2020 adopts a high-voltage power supply featuring novel technology (patent pending) to achieve an ultra-fast polarity switching time of just 15 ms.



Ultra-fast UFsensitivity

Superior sensitivity from UFLC

The newly developed Qarray® Optics achieves superior sensitivity, reproducibility, and linearity.

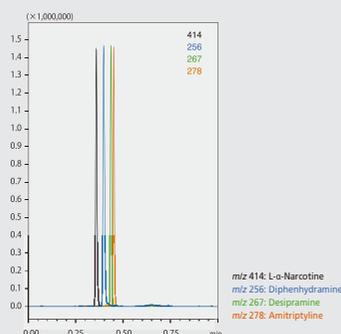


Ultra-fast UFs scanning

15,000 u/sec fast scanning speed

Controls the voltage applied to the Quadrupole according to the scan speed and *m/z*.

This new technology (patent pending) maintains resolution and achieves high ion transmittance even at high scanning speeds.

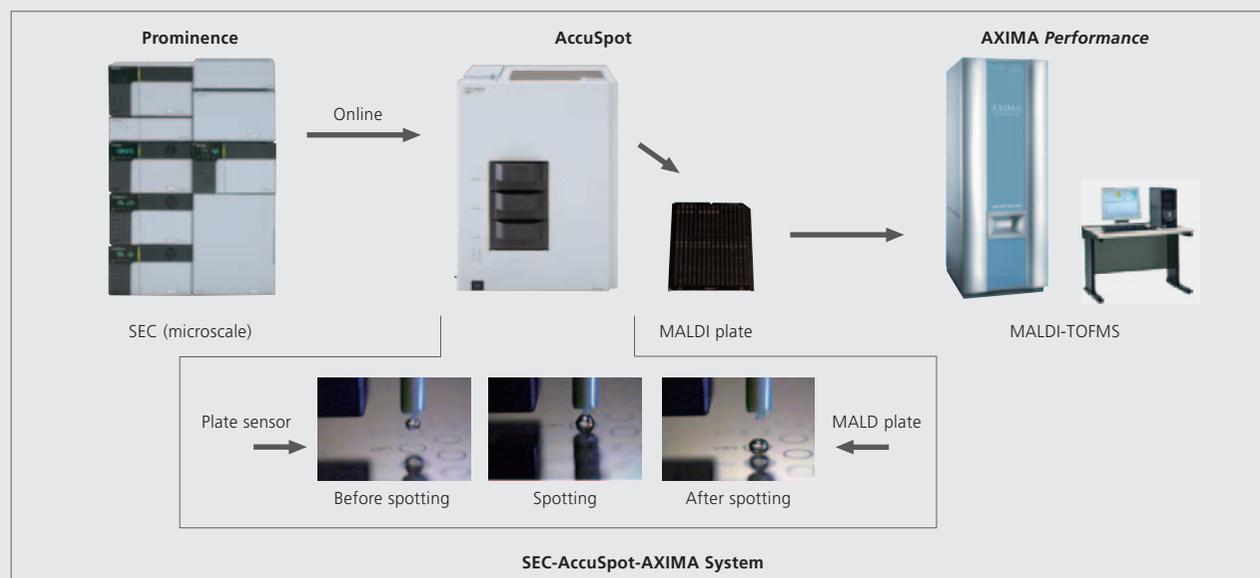


Analysis of Trace Components in Synthetic Polymers Using an SEC-AccuSpot-AXIMA System

SEC-MALDI-TOFMS systems provide a useful method of analyzing trace components in synthetic polymer samples by first using size exclusion chromatography (SEC) to fractionate samples by size and then using MALDI-TOFMS to measure molecular weight information from each fraction. Combining the organic solvent resistant Accuspot system with the AXIMA MALDI TOFMS integrates the entire process from separation by SEC to analysis by MALDI-TOFMS. Therefore, SEC-AccuSpot-AXIMA systems provide a powerful tool for analyzing trace components.

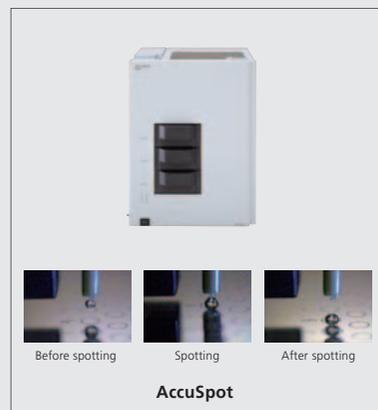
SEC-AccuSpot-AXIMA System

MALDI-TOFMS is widely used as a method for characterizing synthetic polymers. However, if multiple kinds of components are mixed together, the components can mutually inhibit ionization, which results in detection of only the primary components, leaving trace components undetected. The SEC-MALDI-TOFMS method can provide a useful way to avoid that problem by first separating multi-component samples into different fractions. In a typical SEC-MALDI-TOFMS workflow, mixing matrix and cationizing reagents with a large number of fractions for MALDI measurement followed by spotting the fractions onto a MALDI sample plate, is time-consuming and impractical. In contrast, using an SEC-AccuSpot system, which mixes matrix or other reagents with fractions from SEC and successively loads them onto MALDI sample plates, provides a fully integrated process of SEC fractionation and sample spotting for measurement by MALDI-TOFMS. Using the SEC-AccuSpot-AXIMA system reduces the total analysis time to 1/4 the time required previously. Therefore, SEC-AccuSpot-AXIMA systems provide a powerful tool for analyzing trace components.



Features of the Organic Solvent Resistant AccuSpot System

- The AccuSpot is an automatic spotting system that mixes matrix or other reagent with effluent from the LC unit and then successively spots the mixture onto MALDI sample plates.
- Installing an optional GPC compliance kit enables using organic solvents as a mobile phase. GPC Compliance Kit
 - Flow line parts are changed from PEEK to stainless steel or PTFE-based materials.
 - An exhaust fan draws any gases from residual organic solvents out of the instrument and into the fume hood.
- The plate changer function enables continuous spotting of up to 9 MALDI plates.
- Computer interface enables central control of spotting parameters.
- Spotting monitor functionality is included standard
 - The standard-equipped CCD camera allows monitoring of the actual spotting process via the control computer.



Analysis of Plastic Samples Using Thermal Analyzers

Plastics consist of crystallized and non-crystallized areas. In the case of thermoplastics, increasing the temperature causes glass transition and crystallization reactions in the non-crystalline areas, whereas the crystalline areas become soft and flow from melting. As the temperature is increased further, oxidation and decomposition occur. Thermal analysis enables analyzing each of these phenomena easily and quickly. Because these phenomena reflect the various characteristics of plastic products, thermal analysis is useful not only for research and development, but also for quality control.

Shimadzu Differential Scanning Calorimeter DSC-60

- Peak height is about twice as high as previous Shimadzu models.
- Noise level is 1 μ W or less.
- Includes a cooling chamber as standard.

Differential scanning calorimeters provide a useful means of quickly and easily determining the changes in enthalpy and specific heat associated with the first-order transition or relaxation phenomena of substances. In addition to a maximum noise level of 1 μ W and high sensitivity analysis, this new DSC system offers many other unique features as well.



DSC-60

Shimadzu Thermomechanical Analyzer TMA-60

- Multiple types of measurement methods are available to accommodate a diversity of sample shapes.
- Utilizes a new type of displacement sensor with high precision and low drift.
- Accurate automatic length measurement

Thermomechanical analyzers are able to use various types of measurement methods (expansion, tension, and penetration) to evaluate material characteristics of samples with a wide variety of shapes. With features such as an automatic length measurement function and safety mechanisms, this model offers a higher order of high performance and high functionality, combined with ease of operation.



TMA-60

Shimadzu Simultaneous Thermogravimetric and Differential Thermal Analyzer DTG-60

- Utilizes highly sensitive and highly stable balance mechanism.
- Plug-in type DTA detector
- Flow line configuration accommodates a wide range of applications.

Simultaneous thermogravimetric and differential thermal analyzers not only provide higher basic performance levels, they also offer the flexibility to accommodate a wide variety of applications related to interactions between samples and various gas environments. This allows conveniently performing simultaneous measurements for applications ranging from data acquisition to analysis.



DTG-60

Gas Chromatograph

Analysis of Plastic Samples Using a Gas Chromatograph

GC systems are used to analyze volatile components in plastics, such as for testing raw materials, testing elution, analyzing residual monomers, or analyzing residual solvents. Due to its high reliability and ease of analyzing samples, GC systems are well suited to a wide variety of applications, from R&D to process and quality control.

Features of GC-2014

The GC-2014 is a space-efficient multipurpose gas chromatograph featuring the latest state-of-the-art technology.

- Compatible with either capillary or packed columns (or a combination of both)
- Multiple sample injection units and detectors can be installed.
- Space-saving design (40 cm wide)
- A high-performance electronic flow controller (AFC) enables setting column flowrate precisely and easily.
- **Easy to operate via an English language interface displayed on a large LCD screen**
- Smart self-diagnostics function
- Equipped with compact high-sensitivity and high-stability detector



GC-2014

Features of GC-2010Plus

The GC-2010Plus is a capillary gas chromatograph featuring the latest state-of-the-art technology.

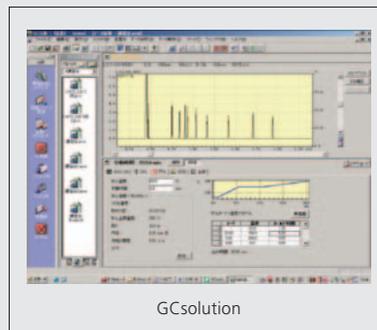
- An electronic flow controller (AFC) offers excellent reproducibility by precisely controlling capillary gas flow at high pressures and flowrate levels required for fast analysis.
- High-performance oven enables rapid heating and rapid cooling.
- Multiple sample injection units and detectors can be installed.
- Electronic flow controller allows setting column and detector flowrates easily.
- Equipped with compact high-sensitivity and high-stability detector
- **Easy to operate via an English language interface displayed on a large LCD screen**
- Smart self-diagnostics function



GC-2010Plus

Features of GCsolution

The GC workstation software "GCsolution" offers extensive functionality, yet is also especially easy to operate. With intuitive operations, assistant bar and data explorer features for providing data management functions, Word-like report functions, and extensive GLP/GMP support functions, the software helps improve analytical productivity. It allows controlling GC-2010 series models, such as the 2014.



GCsolution

Analysis of Plastic Samples Using an SPM

Scanning probe microscope (SPM) is a generic term for microscopes that scan sample surfaces with a microscopic probe to observe their three-dimensional shape or surface properties at high magnifications. SPMs are used for applications such as to observe the surface of plastic materials and products, or analyze their structure or inspect foreign matter. SPMs allow observing three-dimensional surfaces with higher resolution and magnification than optical or electron microscopes. Even insulating samples can be observed directly without modification. SPMs can be used to observe not only shape, but also to measure other physical properties, such as electrical properties, viscoelasticity, and hardness.

Shimadzu Scanning Probe Microscope Features of the SPM-9700

• High Stability and High Throughput

A head-slide mechanism allows sliding the entire optical lever system as an integrated unit. This means samples can be replaced without removing the cantilever and samples can be approached completely automatically, regardless of sample thickness. It also contributes to high throughput by allowing access to samples even during observation. Due to the laser light shining continuously even during sample replacement, stability is higher, which provides clearer and higher quality images.

• Functionality and Expandability Satisfies a Wide Range of Requirements

Standard functionality includes contact, dynamic, phase, lateral force, and force modulation functions. In addition, functionality can be expanded to include electrical current, magnetic force, surface potential, nano-indentation, or nano-thermal analysis functions.

• Ease of Operation Minimizes Distraction Throughout Steps from Observation to Analysis

Easy-to-understand graphical user interfaces (GUI) are used for functions, such as the guidance function and navigation function.

• Wide Variety of 3D Rendering Functions Using Mouse Operations

3D operations, such as rotating or smoothing, can be performed freely in real time. It includes texture and 3D cross sectional profile analysis functions that can be overlaid on height or physical property information.

Environment Controlled SPM Features of the WET-SPM Series

By adding an environment controlled chamber, SPM-9700 series SPMs can be upgraded to a WET-SPM series system. Since the chamber is a glove box system, samples can be prepared (such as cleaving, rinsing, heating, and drying) or replaced within controlled environmental conditions. Depending on the combination of functions and sample operations, some of the environmental controls available include the following.

- Specific gas environment control
- Environmental temperature and humidity control
- Sample heating and cooling
- Blowing gas on samples
- Shining light on samples
- Application of stress on samples



SPM-9700



WET-SPM Series

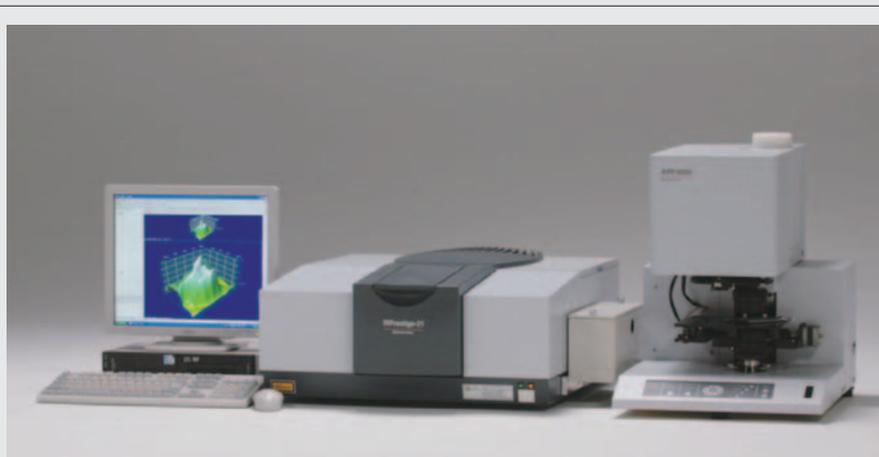
Infrared Microscope System

Analysis of Plastic Samples Using an Infrared Microscope System

Many different types of information can be obtained from plastic samples using FTIR, such as information for checking raw materials, the composition of plastic pellets or powders, bonding modes, isotope ratios, copolymer composition ratios, molecular orientation, and information for analyzing foreign matter or other defects. Using an infrared microscope system, which combines an FTIR spectrophotometer with an infrared microscope, allows obtaining qualitative information about micro areas easily. The AIM View software enables performing all operations via the computer, such as setting the aperture and moving the automatic X-Y stage, and successfully transforms the system into one that is capable of performing highly sensitive infrared microscopic measurements with ease. Using the AIM-MAP mapping software allows capturing the material distribution status of sample surfaces.

Features of the AIM-8800 Infrared Microscope System

- AIM View provides total control.
Microscope operations are completely automated using automatic aperture, X-Y stage, and focus functions. Therefore, sample points, background points, and aperture size settings are remembered and can be reproduced whenever necessary. In addition, an auto-centering function was added, which centers the window image at any arbitrary point that is double-clicked.
- Enables highly sensitive measurements.
Optics have been designed for samples with sides of 50 μm square or less. This is especially beneficial for measuring micro-sized samples and provides higher sensitivity with an S/N ratio of 2600:1 (given a 50 μm square sample and type 1 MCT detector).
- Uses a high-sensitivity maintenance-free MCT detector.
A glass dewar type MCT detector is used, which does not require maintenance. Metal dewar type MCT detectors used by other manufacturers must be re-evacuated every year or two.
- A wide variety of optional products are available.
A variety of optional products, such as an ATR, objective mirrors, diamond cell, and micro manipulator, are available for accommodating a wide variety of applications.
- Use of the optional AIM-MAP mapping software allows capturing the material distribution status of sample surfaces.



Infrared Microscope System
IRPrestige-21 + AIM-8800

Analysis of Plastic Samples Using UV Light

Ultraviolet-visible (near-infrared) spectrophotometers are very effective in evaluating plastics. They are especially useful for evaluating the physical properties of plastic samples, such as when using an integrating sphere to measure transmittance, reflectance, and haze, or by using color measurement software to measure color. They are also widely used to analyze hexavalent chromium for compliance with RoHS or other regulatory requirements.

Features of SolidSpec-3700 (DUV) UV-VIS-NIR Spectrophotometers

- World's first UV-VIS-NIR spectrophotometer equipped with three detectors
- Measures deep-UV regions below 190 nm.
- Extra large sample compartment accommodates a wider variety of samples.

This is the world's first UV-VIS-NIR spectrophotometer to include three detectors. It is ideal for measuring samples in the electrical and optical fields, such as anti-reflection films, high-reflection mirrors, or functionally engineered films. By purging the system with nitrogen gas, DUV models allow measurements in the deep-UV region.

A 3-dimensional optical system and extra large sample compartment allow measuring large samples up to 700 × 560 mm and using an automatic X-Y stage (option) allows automatically measuring multiple points on samples up to 300 × 300 mm.



SolidSpec-3700 (DUV)

Features of UV-2600/UV-2700 UV-VIS Spectrophotometers

- UV-2600: Single-monochromator model that offers a wide wavelength range and low noise
- UV-2700: Double-monochromator model that achieves ultra-low stray light levels

The key feature of the UV-2600 is its wavelength measurement range. If an ISR-2600Plus integrating sphere attachment is used, this system can measure wavelengths from 220 nm to 1400 nm, which includes the near-infrared region, extending its usefulness to an even wider range of applications. It can also be used to measure solar cell materials and various types of glass samples. The UV-2700 can achieve ultra-low stray light levels, with transmittance of 0.000001% (1/10 of a billionth) and an absorbance range up to 8. Due to a dramatically higher precision level, it is able to measure higher absorbance levels. This means highly concentrated samples can be measured without pretreatment, which not only saves the time and trouble of diluting samples, but also means it can be used to evaluate transmission characteristics of polarizing films.



UV-2600/2700

Features of UVmini-1240 UV-VIS Spectrophotometers

- Compact design equivalent to size of **A3 (297×420 mm)** paper
- Easy to operate
- Measures wavelengths from 190 to 1100 nm.

The UVmini-1240 is capable of everything from simple colorimetric analysis to sophisticated quantitative analysis and spectral measurements. All systems are equipped standard with photometric, spectral, and quantitative measurement modes. The compact **A3 (297×420 mm)** size saves space. Measurement results are displayed on a 6-inch backlit LCD screen. Functionality for printing and output via an RS-232C cable is included standard, which also allows printing a copy of screen contents to a standard printer. Measurement results can be saved in internal memory or saved in an optional data pack. Furthermore, UV Data Manager software (option) is also available as a tool for loading data to the computer. Of course, with a wide selection of available accessories, it can accommodate a diverse variety of applications.



UVmini-1240

Evaluation of Mechanical Characteristics of Plastic Products Using Autograph

Due to their various mechanical characteristics, plastic products are used in an extraordinarily broad range of applications. The most fundamental of mechanical characteristics is the strength necessary to ensure safety and reliability. The Autograph series are also able to obtain information about many other aspects of products, such as elongation, displacement, and energy, providing important information not only for controlling product quality, but also for developing new products.

Features of Autograph AG-X Series

- **New design introduces a new era (loading frame and software)**

The loading frame is designed with a sophisticated look to match laboratories and an interface located on the front side for easier operability. The specialized TRAPEZIUM X software features a new design that provides a perfect balance between aesthetics and ease-of-operation.

- **Stress and strain controls enable precision control**

An autotuning function enables highly precise stress or strain control by simply setting the testing speed, which eliminates the need to specify gain or other parameter settings. Eight times more precise control resolution than previous Shimadzu models ensures accurate control even at low speeds (perfect for constant force control used to test films, rubbers, and fibers).

- **World's first to offer test force measurement precision to 1/1000**

The high-precision model offers measurement precision to within 0.5 % of the indicated value over a wide range of applied loads, from 1/1 to 1/1000 of the load cell capacity rating. This means load cells can be exchanged less often (details depend on individual specifications).

- **Ultra fast 0.2 msec sampling**

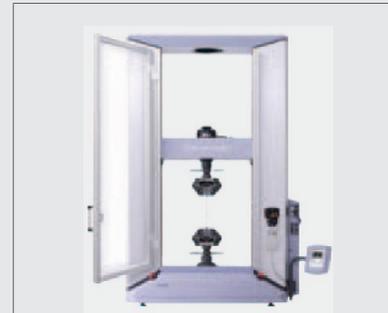
During tests, measurement data can be acquired at sampling intervals as short as 0.2 msec. This allows evaluating composite materials and performing highly reliable measurements.

- **Self-check function keeps machine in optimal condition**

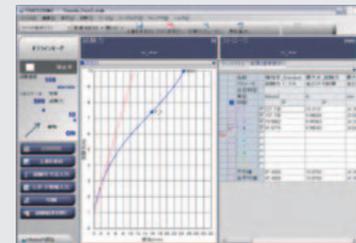
The system status can be confirmed via the self-check function that runs automatically during startup or via the checking functions that periodically run in a guidance format. The function that automatically notifies users of scheduled maintenance is also able to keep track of validation dates and other information.

- **Easily accommodates a variety of tests and improves test efficiency**

A color touch panel display (option) allows specifying test parameter settings and running tests without a computer. It allows displaying graphs during testing. Test parameter settings configured on the computer can be saved in USB memory, which can then be used to perform tests. By saving test results automatically in USB memory and plugging that into the computer, identical operations can be performed as when the computer is connected to the testing machine.



Autograph AG-X Series



Specialized TRAPEZIUM X Software

A Variety of Accessories Expand the Range of Applications

The Autograph series offers an extensive selection of accessories available for accommodating a wide range of specimens and objectives. By selecting the optimal accessories, such as loading jigs (grips and so on) that apply the desired force to specimens, sensors that accurately measure the displacement (such as elongation and deflection) at specific locations, or atmosphere conditioning systems for controlling the testing environment (such as temperature and humidity), it is possible to configure systems optimized for testing objectives.



Thermo Chamber

Grips and Extensometer

Evaluation of Endurance and Impact Characteristics of Plastic Products Using Servopulser/Hydroshot Testing Machines

To ensure reliability and safety under actual usage conditions, it is essential to understand and control not only basic static strength, but also endurance (with respected to repeated stresses) and impact resistance characteristics. Servopulser fatigue testing machines are able to efficiently apply precise repeated loads and reproduce various load waveforms experienced in actual operation. Hydroshot high-speed impact testing machines can obtain failure characteristics data at speeds up to 20 m/sec.

Features of Servopulser

- **Loading capacity and loading mode selectable based on a test object**
The hydraulic configuration is able to repeatedly apply large loads (up to 300 kN for standard models) at high speed, which allows fatigue testing of almost any material or mechanical part.
- **Stress and strain controls enable precision control**
Closed loop servo control enables reproducing loading waveforms accurately and faithfully. With fundamental cycle waveforms as well as random and actual operating load waveforms, simulation testing can be supported as well.
- **Select from two available controllers**
Select either the compact 4830 controller or the 4890 controller, which allows using various data analysis modes. Connected to a computer, these allow seamlessly performing all testing tasks via the computer, including configuring test parameter settings, executing tests, and analyzing data.
- **Extensive assortment of accessories**
A wide assortment of accessories, such as test jigs (grips and so on), various external sensors (to measure elongation and displacement), and environment control devices (such as high/low temperature and high/low humidity), are available for configuring an evaluation system that is optimized for given objectives.
- **Electromagnetic and pneumatic systems are also available**
For smaller loading capacity requirements, electromagnetic (Micro-Servo) and pneumatic (Air-Servo) systems are also available as an alternative to hydraulic actuation. These models offer lower environmental impact and are ideal for fields such as electronics.
- **Also capable of multi-axis testing**
Multiaxial systems (maximum 4 axes) can also be configured, which are ideal for testing the endurance of actual parts, such as automobile components. Features include an interaxial interference correction function based on transfer functions and a resonant frequency tracking function.



Tabletop Servopulser (Hydraulic)



4830 Controller and Operation Computer

Features of Hydroshot

- **Hydraulic actuation enables accurate speed control**
Hydroshot testing machines feature accurate flowrate control of pressurized hydraulic oil and enable high capacities (maximum 10 kN) and high loading speeds (maximum 20 m/sec).
- **Two types available – puncture or tension**
The product line includes two types, the HITS-P10 model for puncture testing and the HITS-T10 for tensile testing.
- **Accurate measurements**
A test force detector (load cell) designed to minimize reflection of vibration and stress waveforms during high-speed loading and a crosshead displacement detector ensure accurate data acquisition. A non-contact type chuck displacement gauge (for tensile testing) is effective in measuring the amount of deformation in the area closest to the specimen.
- **Extensive assortment of accessories**
An extensive line of accessories are available to configure the optimal system, such as various light-weight grips best-suited to the given specimen, approach jigs for tensile testing, and thermostatic chambers to control the temperature of the testing atmosphere.
- **Specialized operating software**
All machines include software designed specifically for operating Hydroshot machines. This ensures that all operations, from setting test parameters to organizing data can be performed as simply and safely as possible.



Hydroshot HITS-T10 High-Speed Impact (Tensile) Testing Machine

Industrial X-Ray Inspection System

Observing the Interior of Plastic Products Using Industrial X-Ray Inspection (Fluoroscopy/CT) Systems

Light and strong fiber reinforced plastics (FRP) are not only used in construction materials and sport equipment, but also used to reduce energy consumption in automotive and aerospace industries. Functionally engineered polymer materials are used in next-generation batteries (such as fuel cells, solar cells, and Li-ion batteries), which have been attracting significant interest. In many cases, these materials have increasingly complicated structures with characteristics that cannot be adequately evaluated using a conventional singular approach of evaluating physical properties. Therefore, there has been a growing interest in 3-dimensional data analysis methods. This example offers a method of analysis that uses a CT system to observe samples with X-rays.

Features of Industrial X-Ray Inspection (Fluoroscopy/CT) Systems

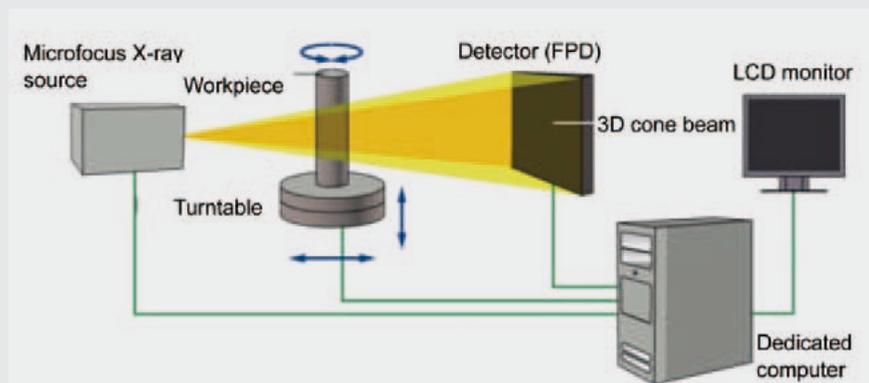
- Obtains ultra high-speed and high-resolution (micron level) CT images (cross sections).
- A line of three models - small, medium and large, allows selecting the best model for the intended application.
- The user interface allows even first-time users to operate the system easily. For example, the manipulator can be operated by simply moving the mouse pointer on the fluoroscopy screen. In addition, a large opening allows loading even bulky samples with ease.
- Since no troublesome calibration operations are required, high-quality images can be obtained easily by anyone.
- Image acquisition can be started immediately while irradiating the sample, by simply deciding the magnification rate.
- System functionality can be expanded to include 3D dimensional measurement or various other optional (software) analytical functions.
- Systems can be used to measure a wide variety of samples, from composite materials, electrical/electronic devices/parts, mechanical parts, and plant and biological samples.



inspeXio SMX-90CT Benchtop Microfocus X-Ray CT System



inspeXio SMX-225CT Microfocus X-Ray CT System



Operating Principle of X-Ray CT Scanning

Analysis of Plastic Samples Using an AA Spectrophotometer

Atomic absorption spectrophotometers are used to analyze trace hazardous inorganic elements in plastic, in particular cadmium, lead, and arsenic. If there are only a few target elements being measured, this allows measuring them efficiently at low cost. Plastic samples are normally pretreated by adding acid to thermally decompose them before measuring. Some plastics can be simply dissolved in an organic solvent before measurement. If target elements are present in higher concentrations, flame atomization is used, which allows a shorter measurement time. However, when measuring trace quantities, selecting the furnace measurement method allows analyzing the samples more efficiently.

Shimadzu Atomic Absorption Spectrophotometers Features of AA-7000

- **System configurations advancing together with needs**
Compact dedicated flame models can be converted to a dual atomizer system that can automatically switch between flame/furnace modes. Dedicated furnace models are also available.
- **Fully featured flame analysis**
A newly developed 3-dimensional double-beam optical system and sturdy hardware provide outstanding stability.
- **Advance safety technology**
The system features many safety mechanisms, such as the world's first automatic flame extinction via flame vibration sensor, multimode automatic gas leak check, and use of flame resistant materials.
- **World-class high-sensitivity furnace**
Lower limit of detection performance was improved by reducing noise with state-of-the-art optics. Stability is also improved by digital temperature control and digital gas control.
- **Dual atomizer system**
(automatic flame/furnace mode switching function)
Automatically switches the atomization unit by software operation. A newly developed drive mechanism cuts the switchover time in half from the previous model.
- **Multifunctional autosampler**
A single autosampler supports both flame and furnace analysis.
Commercial pipette tips can be used for nozzles in the furnace mode.
A rinse port in the overflow mechanism ensures nozzles are rinsed properly.
- **Features easy to understand and use WizAArd software**
Extensive display features and highly flexible settings
Supports system control and precision control and includes hardware validation software.



AA-7000 Dual Atomizer System



WizAArd High Functionality Software



AA-7000 Flame Model

AA-7000G Furnace Model

Multitype ICP Emission Spectrometer

Analysis of Plastic Samples Using an ICP Spectrometer

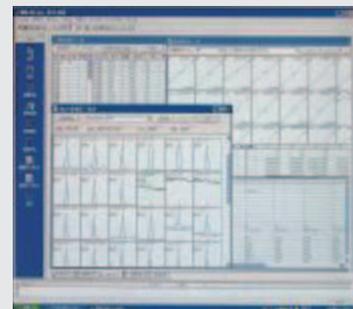
ICP emission spectrometers are used to analyze trace hazardous inorganic elements in plastic, in particular cadmium, lead, and arsenic. Plastic samples are normally pretreated by adding acid and thermally decomposing before measuring. This system enables simultaneously measuring multiple elements with high sensitivity and high precision. Furthermore, it also enables qualitative analysis to confirm the presence of most inorganic elements in samples.

Shimadzu Multitype ICP Emission Spectrometers Features of ICPE-9000

- High throughput**
 Uses an Echelle spectrometer capable of high-speed measurement and large high-resolution CCD detector.
- Equipped with Mini Torch**
 The mini torch offers equivalent sensitivity to a standard torch, but consumes half the argon gas. The vertical orientation minimizes contamination and clogging, which is especially reassuring for concentrated samples.
- Vacuum spectrometer**
 This is the first ICP emission spectrometer with a semiconductor detector that is equipped with a vacuum spectrometer. No high-purity gases are necessary for purging. This not only reduces operating costs, but also provides superior spectrometer stability.
- Even the first sample is easy to analyze with ICPEsolution software**
 Includes qualitative database calibration functionality.
 Method Development Assistant automatically prepares measurement parameters.
 Includes Automatic Wavelength Selection System.
 Method Diagnosis Assistant checks for causes of errors.



ICPE-9000



ICPEsolution High Functionality Software

Multiple displays of measurement results

Conventional Echelle Instrument measurement

Element and Wavelength	Sample A	Sample B
1. Cd 226nm	5.6	78.5
2. Cd 228nm	37.9	7.1

Units: ppm

Multiple results with different values are displayed since the presence of interference cannot be determined. Depending on the device, a simple average of these values may also be displayed. An analyst's skill is required for a correct evaluation.

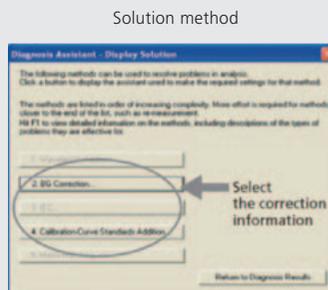
Display of only a single measurement result

ICPE-9000

Element and Wavelength	Sample A	Sample B
1. Cd 226nm	5.6	—
2. Cd 228nm	—	7.1

Units: ppm

Example of Automatic Wavelength Selection System



Example of Problem-Solving Using Method Diagnosis Assistant

Evaluation of Plastic Materials

Evaluation of Raw Materials

Product Evaluation

Product Information

Energy Dispersive X-Ray Fluorescence Spectrometer

Product Information

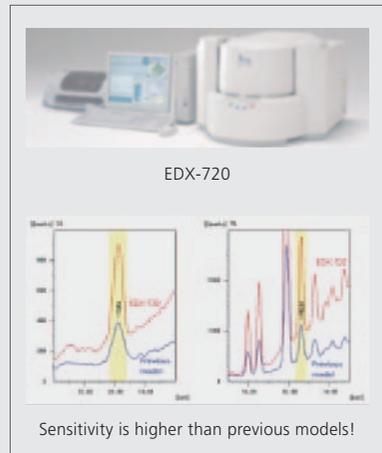
Product Evaluation

Analysis of Plastic Samples Using an EDX Spectrometer

When samples are irradiated with X-rays, they emit fluorescent X-rays that are characteristic of the elements contained in the sample. EDX spectrometers enable measuring samples rapidly for controlling the content of various additives or for analyzing sample impurities or defects. More recently, due to their convenience, they have become widely used for screening five elements regulated by the RoHS directive and four elements regulated by the ELV directive.

Features of EDX-720 Energy Dispersive X-Ray Fluorescence Spectrometers

- Allows directly and non-destructively measuring various forms of metals, plastics, powders, liquids, and other materials. biological samples.
- Measurements require only specifying the target area by viewing the CCD camera image (optional).
- Rapidly measures the five RoHS elements and four ELV elements. A specialized primary filter is also included standard.
- Also can be used for general material analysis, defect analysis, or coating thickness analysis.

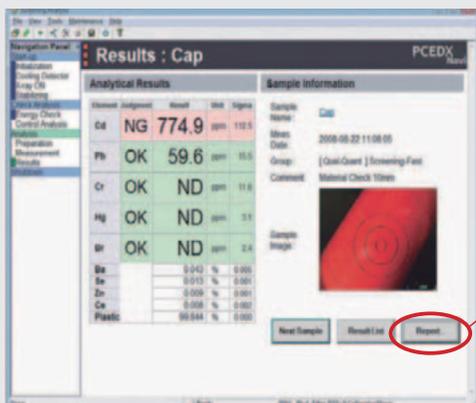


Features of EDX-GP/LE Energy Dispersive X-Ray Fluorescence Spectrometers

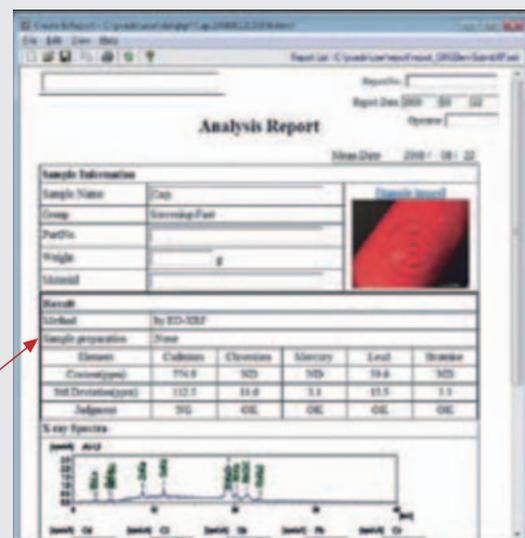
- Includes screening analysis software with simplified operations.
- Also can be used for halogen-free analysis by using a specialized filter to increase sensitivity.
- Features a large sample compartment.
- Equipped standard with sample observation function (CCD camera).



EDX-GP/LE



Screening Analysis Software



Create reports with a single click!

Evaluation of Plastic Materials

Evaluation of Raw Materials

Product Evaluation

Product Information



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