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Differences Between IRSpirit-L and IRSpirit-T

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1. Operating Principal of a Thermal Detector

In October 2017, Shimadzu launched the compact IRSpirit FTIR spectrophotometer. The IRSpirit product line-up contains two models that differ by detector type: the standard IRSpirit-L that comes with an LiTaO₃ detector (hereinafter "LTO detector"), and the high performance IRSpirit-T that comes with a DLATGS detector. Both detectors are classified as thermal detectors that use the pyroelectric effect to convert fluctuations in heat into electrical signals. Materials that exhibit this pyroelectric effect are called pyroelectric elements. Common products that utilize pyroelectric elements include lighting linked to motion detectors that turn on automatically upon detection of movement, and microwaves that use pyroelectric elements to detect the heat from food. The thermal time constant is one of the indicators of pyroelectric element performance, and the pyroelectric elements used in these everyday applications have a high thermal time constant that allows them to detect thermal changes within the order of several hundred milliseconds. In FTIR spectrophotome-

try, thermal changes arising from wave interference, caused by movement of the interferometer moving mirror, are converted to electrical signals. The detectors have a lower thermal time constant to detect temperature changes within the order of tens of milliseconds. The thermal time constant is normally dependent on the thickness of a blackened film on the surface of the pyroelectric element, where a thicker film increases the time of thermal conduction that increases the thermal time constant. Although a thinner film gives a lower thermal time constant, it also leads to reflection of infrared light at the element surface and electrode that are in contact with the pyroelectric element, which causes the issue of interference. For this reason, controlling the thickness of the blackened film is important in the manufacture of pyroelectric elements. The change in electric charge that occurs during detection of heat by a pyroelectric element is shown in Fig. 1.

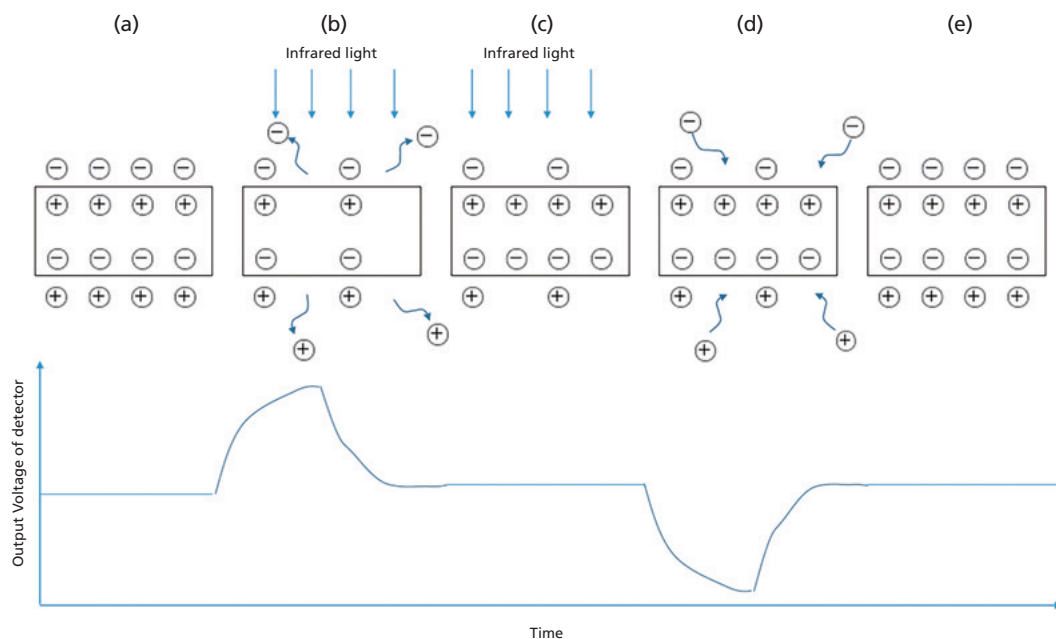


Fig. 1 Charge Behavior and Output Voltage of Pyroelectric Element

The centroids of positive and negative charges within a pyroelectric element crystal do not coincide, but exhibit spontaneous polarization. Spontaneously polarized charges exist at the surface of the crystal, and neutral conditions are created by the attachment of charged particles from the atmosphere to the crystal surface. The resulting voltage created by the pyroelectric element is the offset voltage (Fig. 1 (a)). The position of these positive and negative charges is temperature dependent. Changes in temperature cause a change in the polarization conditions, which in turn changes the voltage output by the pyroelectric element (Fig. 1(b)). When this is followed by emission of a constant amount of infrared light, the voltage output by the

pyroelectric element then returns because the temperature remains constant (Fig. 1(c)). When the infrared light is blocked, the spontaneous polarization is changed and the sensor output switches to the opposite direction of the conditions shown in (b) (Fig. 1(d)). When the temperature of the pyroelectric element does not change, the positive and negative charges also remain unchanged, and there is no output signal. With an interferometer, temperature is varied by movement of the moving mirror, which generates an output signal. This output signal is composed of various overlapping sine waves, and Fourier transformation can be used to resolve these frequencies and obtain an intensity spectrum of infrared light.

2. Differences in the Infrared Detector Element

This section will introduce the differences between the IRSpirit detectors. The LTO detector used in IRSpirit-L is formed from lithium tantalate crystal, and the DLATGS detector used in IRSpirit-T is formed from triglycine sulfate (TGS) crystal doped with deuterated L-alanine. The DLATGS detector is also used as the standard detector in higher-end FTIR systems, including IRAffinity-1S and IRTTracer-100. The LTO detector newly introduced to IRSpirit-L is highly temperature stable, and the crystal used can be created at a relatively low cost compared to the DLATGS detector. Due to the reasonable price, LTO detectors are used in Surface Acoustic Wave (SAW) devices* that are key parts of mobile phones and other wireless communications equipment. The detecting performance of LTO detectors and DLATGS detectors is dependent on pyroelectric performance. Since only a very small current can be obtained from a pyroelectric element, it is normal to

include a voltage mode circuit that connects a high-impedance resistance with a junction FET. The voltage sensitivity R_V of the sensor in this mode of operation is shown by the following equation.

* What is a SAW device?

Surface Acoustic Wave (SAW) devices have a filtering function that uses the surface waves that propagate through a piezoelectric substrate. Piezoelectric substances are materials that deform under application of voltage, and also generate voltage from surface charge under application of force (the piezoelectric effect). All ferroelectric substances that exhibit the pyroelectric effect also exhibit the piezoelectric effect. SAW filters convert a high-frequency signal introduced as an electrical signal into a surface wave of several μm wavelength based on the piezoelectric effect of a piezoelectric substrate. This surface wave is propagated through the piezoelectric substrate and output once again as an electrical signal after filtering for the desired surface wave frequency. For this reason, SAW filters always produce an electrical signal as an output.

$$R_V = \frac{\eta P}{C_V \epsilon \epsilon_0} \frac{1}{A} \frac{1}{\omega}$$

Where η is the emissivity of the incident plane, P is the pyroelectric coefficient, C_V is specific heat at constant volume, ϵ_0 is magnetic permeability of a vacuum, ϵ is relative permittivity of a pyroelectric material, A is light-receiving area, and ω is angular frequency of incident light. When frequency is taken as f ,

$$\omega = 2 \pi f$$

Where voltage sensitivity R_V is dependent on frequency. Since voltage sensitivity decreases as frequency increases, increasing the speed of the interferometer moving mirror reduces the voltage sensitivity. The equation shown below gives a performance index for the voltage sensitivity of pyroelectric elements that can be used to compare voltage sensitivity.

$$F_V = \frac{P}{C_V \epsilon}$$

The voltage sensitivity evaluation indices of an LTO detector and DLATGS detector are as shown below.

Table 1 Pyroelectric Performance of Each Element

| Pyroelectric Element | Curie Temperature (°C) | Relative Permittivity | Pyroelectric Coefficient (C/cm ² /K) × 10 ⁻⁸ | Voltage Evaluation Index (C·cm/J) × 10 ⁻¹⁰ |
|----------------------|------------------------|-----------------------|--|---|
| LTO | 618 | 54 | 2.0 | 1.3 |
| DLATGS | 62 | 35 | 4.0 | 4.6 |

Based on the voltage evaluation index, the sensitivity of DLATGS is around 3.5 times better than LTO. Based on the amount of element noise in the two sensing elements used in Shimadzu's IRSpirit line-up, DLATGS noise is around 1.3 times higher than LTO. Therefore, the S/N ratio of DLATGS is around 2.5 times higher than that of LTO. Although there is a 4-fold difference in voltage sensitivity between the elements, equivalent infrared intensities are displayed in the software due to an amplifier board that provides a 4-fold gain for the LTO detector.

DLATGS polarization also varies greatly with ambient temperature changes as the Curie temperature of DLATGS is close to room

temperature. Because these changes to polarization affect the data being collected, DLATGS detectors require temperature control (IRSpirit DLATGS detectors are equipped with a temperature control function). LTO is not affected by ambient temperature changes due to a Curie temperature of around 600 °C, and LTO detectors do not require temperature control.

Exposure to atmosphere causes performance degradation of the detector element in both LTO detectors and DLATGS detectors. In IRSpirit models, detector elements are enclosed in a box that shields it from the atmosphere but allows incident infrared light through an aperture plate (aperture plate material is KRS-5).

3. Differences in Actual Measured Value

This section will confirm differences in actual measured value obtained from IRSpirit-L and IRSpirit-T upon analysis of the same sample. Commercially available plastic bags (polyethylene) were used as the sample, and QATR-S, an Attenuation Total Reflectance (ATR) accessory designed for IRSpirit, was used to take measure-

ments by single reflection ATR. A photograph of the plastic bags is shown in Fig. 2.

Measurement conditions are shown in Table 2, and results from IRSpirit-L and IRSpirit-T measurements are shown in Fig. 3.

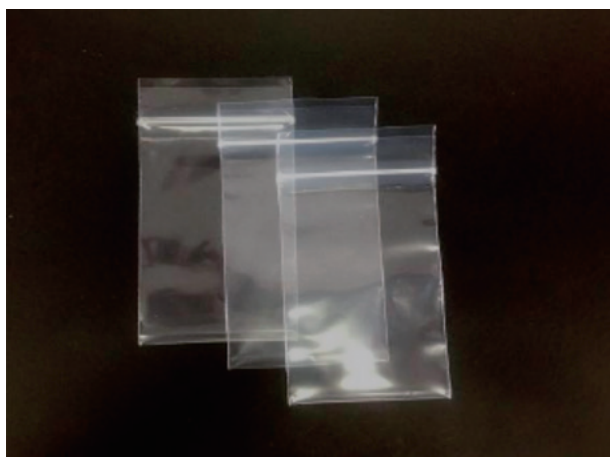


Fig. 2 Plastic Bags

Table 2 Measurement Conditions

| | |
|------------------------|-----------------------------|
| Equipment | : IRSpirit-L and IRSpirit-T |
| Resolution | : 4.0 cm ⁻¹ |
| Number of Measurements | : 20 |
| Apodization Function | : Square-Triangle |
| Accessories | : QATR-S (prism: diamond) |

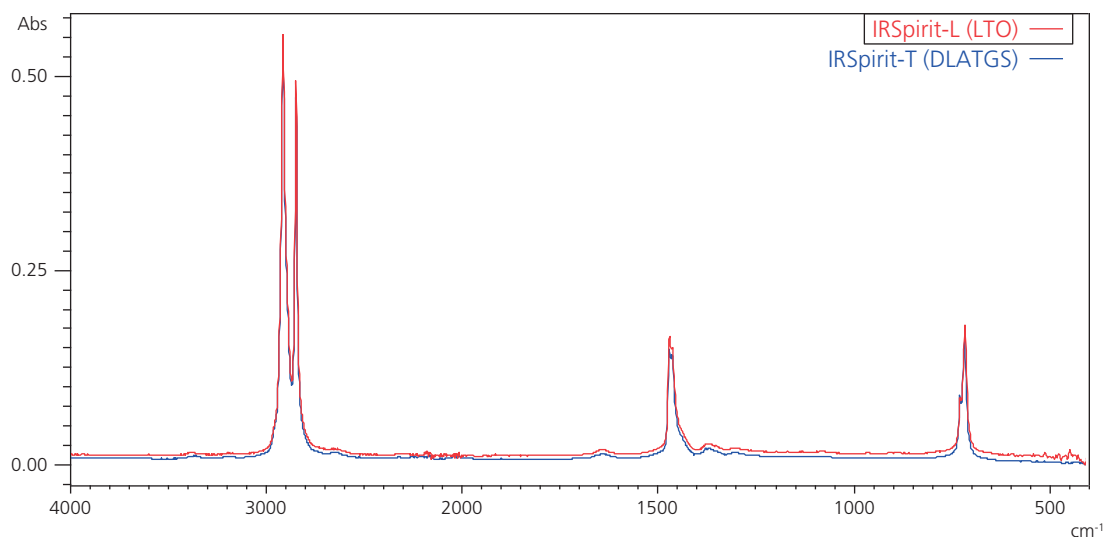


Fig. 3 ATR Spectra of Plastic Bag (Red Line: IRSpirit-L, Blue Line: IRSpirit-T)

Fig. 3 shows no difference between the systems. The results shown in Fig. 3 have been expanded and are shown in Fig. 4 across a reduced wavenumber range (1,200 to 600 cm^{-1}) that is unaffected by atmospheric water vapor or carbon dioxide.

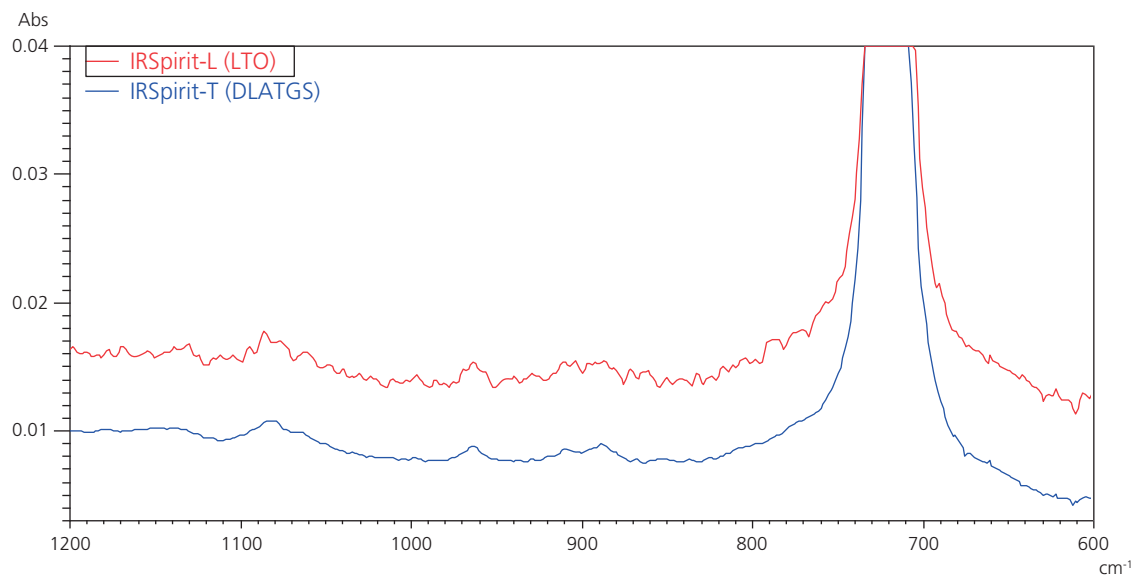


Fig. 4 Expanded View of Fig. 3 (1,200 to 600 cm^{-1})

The difference between systems not apparent in Fig. 3 is distinguishable in Fig. 4. It shows the baseline noise of IRSpirit-T is very small compared to IRSpirit-L. IRSpirit-T is recommended for use when an analyst wants to analyze small peaks with good sensitivity (the S/N ratio of IRSpirit-L can be improved to a degree by increasing the number of measurements, but this also increases the measurement time).

Noise is generally improved by increasing the number of measurements, where an n -fold increase in S/N ratio requires an

n^2 -fold increase in measurements. As mentioned earlier, DLATGS detectors are around three times more sensitive than LTO detectors, so to achieve the same S/N ratio as a DLATGS detector, an LTO detector would, in theory, require nine times the number of measurements. This may not always be the case, however, as the sensitivity difference is also dependent on temperature control in the DLATGS detector, transmittance of the aperture plate, and the throughput of accessories.

4. Summary

We have introduced actual measured values and described differences between the detectors used in IRSpirit-L and IRSpirit-T. Equipped with an LTO detector, IRSpirit-L can detect small peaks to a degree by increasing the number of measurements, though the analyst should use IRSpirit-T if they want to acquire

data with good sensitivity within a short period of time, and also to analyze very small contaminants (the high-end IRTracer-100 model also allows detection of extremely small peaks at the sub-mAbs scale).

Features of the "Contaminant Library"

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The "Contaminant Library" is a unique library of infrared spectra developed by Shimadzu and intended to support contaminant analysis performed by FTIR users. This article describes the features of this library, along with example applications and points to note.

1. Introduction

A wide variety of contaminants find their way into products, from rubber to packing materials and different types of seal materials to metal. This contamination can occur due to deteriorating manufacturing facilities and installations leading to a defective component, or a part of a worker's body or clothing mixing into the product. It is also not uncommon to have cases in which the user (consumer) is responsible for introducing a contaminant. When a complaint of a contaminant found in a product is received, the cause of the contamination must be quickly ascertained, this information provided to the user, and action taken to prevent recurrence. Although infrared spectrophotometers (FTIR) and X-ray fluorescence (XRF) spectrometers are used to perform qualitative analysis of contaminants, in this article we will describe using Shimadzu's original "Contaminant Library" to perform more effective contaminant analysis.

2. What is the "Contaminant Library"?

The contaminant library is a spectral library intended for use in contaminant analysis and created with the cooperation of water-supply corporations and food manufacturers. The library contains information on samples collected in the real world as actual contaminants, and includes data such as the infrared spectrum of the contaminant, its color, shape and hardness. The library also includes X-ray fluorescence profiles (PDF files), which allows the library to be used as a reference on inorganic compounds. Table 1 shows an excerpt of the information contained in the library. The features and benefits of this library will be explained below.

Table 1 Example Information Held in Contaminant Library (Excerpt)

■ Rubber ■ Polymer ■ Bio related ■ Inorganic

| | Name | Detailed Information | | | Metal Component Estimated by EDX Analysis |
|---------------------------------------|----------------------------------|--|---|--|---|
| | | FTIR Qualitative Analysis Result (Materials) | EDX Quantitative Analysis Result (Major elements) | Color/Shape/Hardness/Metallic luster | |
| Water Supply Related Foreign Matter | 083_Water meter packing1_Outside | Acrylonitrile butadiene rubber(NBR),Zinc stearate | S,Zn | Color;Black Shape;Rubber/Fragment Hardness;Soft Metallic luster;No | |
| | 084_Water meter packing1_Inside | Acrylonitrile butadiene rubber(NBR) | S,Zn | Color;Black Shape;Rubber/Fragment Hardness;Soft Metallic luster;No | |
| | 087_Coating of inner wall_1 | Polystyrene(PS),Acrylic resin | Al,Si | Color;Brown Shape;Fragment Hardness;Soft Metallic luster;No | |
| | 088_Corrosion inhibitor | Polyvinyl acetate(PVAc),Hydrated magnesium silicate(TALC,Mg3Si4O10(OH)2) | Si,Ca,Ti,Mg | Color;Gray Shape;Fragment Hardness;Soft Metallic luster;No | |
| | 107_Coating agent | Epoxy resin,Hydrated magnesium silicate(TALC,Mg3Si4O10(OH)2) | Si,Mg,Fe,Cl | Color;Black Shape;Fragment Hardness;Hard Metallic luster;No | |
| | 103_Biofilm_1 | Cellulose,Mold | below 1% | Color;White/Black Shape;Biofilm Hardness;Soft Metallic luster;No | |
| | 139_Mold | Protein,Silicate | below 1% | Color;Brown Shape;Mold Hardness;Soft Metallic luster;No | |
| | 091_Scale_1 | Calcium carbonate(CaCO3) | Ca | Color;White Shape;Powder/Cluster Hardness;Brittle Metallic luster;No | |
| | 186_Disposal ash | Silicate,Phosphate | Si,Ca,P,Fe,Al,Mg,K,Ti,Zn,Ba | Color;Brown Shape;Sand/Cluster Hardness;Soft Metallic luster;No | |
| | 316_Iron rust_4 | Iron(III) hydroxide(Fe(OH)3),Silicate | Fe,Si,S | Color;Brown Shape;Iron rust Hardness;Hard Metallic luster;No | Iron |
| Food Related Foreign Matter | 356_Elastic | cis-Polysoprene rubber | below 1% | Color;Green Shape;Rubber Hardness;Soft Metallic luster;No | |
| | 386_Cluster of Nylon fiber | Polyamide,Polyester,Cooking oil(Triacylglycerol) | below 1% | Color;Black Shape;Fiber Hardness;Soft Metallic luster;No | |
| | 412_Cluster of sugar | Lactose | below 1% | Color;White Shape;Cluster Hardness;Hard Metallic luster;No | |
| | 428_Cluster of starch | Starch,Cooking oil(Triacylglycerol),Protein | Cl,Na | Color;Brown Shape;Cluster Hardness;Hard Metallic luster;No | |
| | 342_Plant material1 | Cellulose,Vegetable fat(Triacylglycerol) | Cl,Na | Color;Brown Shape;Cluster Hardness;Soft Metallic luster;No | |
| | 362_Bone particle_white | Calcium phosphate,Protein | Ca,P,S | Color;White Shape;Stick Hardness;Hard Metallic luster;No | |
| | 402_Nail | Keratin | S | Color;White Shape;Fragment Hardness;Hard Metallic luster;No | |
| | 370_Stone1 | Silicate | Si,Al,Fe,K,Ti,Mg,Na | Color;Black Shape;Fragment Hardness;Hard Metallic luster;No | |
| | 404_Iron needle | Adsorbate on metal surface | Fe,P | Color;Black Shape;Fiber Hardness;Hard Metallic luster;No | Iron |
| | 454_Stainless steel_1 | Adsorbate on metal surface | Fe,Cr,Ni,Mn | Color;Silver Shape;Metal Hardness;Hard Metallic luster;Yes | Stainless |
| Commercial Water Supply Related Parts | 005_Pipe 19mm packing_Outside | Styrene butadiene rubber(SBR),Calcium carbonate(CaCO3),Polymethyl methacrylate(PMMA) | Ca | Color;Black Shape;Rubber/Ring Hardness;Soft Metallic luster;No | |
| | 006_Pipe 19mm packing_Inside | Styrene butadiene rubber(SBR),Calcium carbonate(CaCO3) | Ca | Color;Black Shape;Rubber/Ring Hardness;Soft Metallic luster;No | |
| | 194_Pipe packing_Red | Polyvinyl chloride(PVC) | Cl | Color;Red Shape;Resin/Ring Hardness;Hard Metallic luster;No | |
| | 200_Single packing_Outside | Ethylene propylene diene rubber(EPDM),Silicate | Si,Zn,S | Color;Gray Shape;Rubber/Ring Hardness;Soft Metallic luster;No | |
| | 001_String seal | Polytetrafluoroethylene(PTFE) | F | Color;White Shape;Fiber Hardness;Soft Metallic luster;No | |
| | 026_Hardened adhesive | Vinyl chloride-vinyl acetate copolymer,Cyclohexanone | Cl | Color;Transparency Shape;Cluster Hardness;Soft Metallic luster;No | |
| | 017_Handle_packing_13mm_Metal | Fatty acid ester,Carboxylate,Cellulose(Adsorbate on metal surface) | Cu,Zn | Color;Gold Shape;Ring Hardness;Hard Metallic luster;Yes | Brass |

2.1 Rich Variety of Spectra on Contaminants and Components Found in Piping and Flow Paths

The library includes spectra for fastening components in piping and flow paths, such as rubber and packing materials that may become contaminants, as well as actual contaminants collected by water-supply corporations. Many contaminants and components are not made of a single constituent, but are a mixture of multiple constituent materials. For this reason, many of the spectra collected in the library are of a mixture of constituents. A spectral search of a spectral library containing spectra of single-material samples will probably give very few hits and make contaminant identification very difficult. Meanwhile, Shimadzu's contaminant library contains a number of spectra that will match samples over almost the entire spectral waveform.

Fig. 1 shows the results of analyzing commercially available rubber packing material and a subsequent spectral search of the contaminant library. The main constituent of the rubber packing material is presumed to be acrylonitrile butadiene rubber (NBR), with calcium carbonate and stearate also present as additives. The spectral waveform of the analyzed rubber packing material and of the rubber component used in creating the contaminant library are very similar, despite being procured from two completely different places at entirely different times. This demonstrates the utility of the contaminant library for identification of contaminants that contain a mixture of constituents.

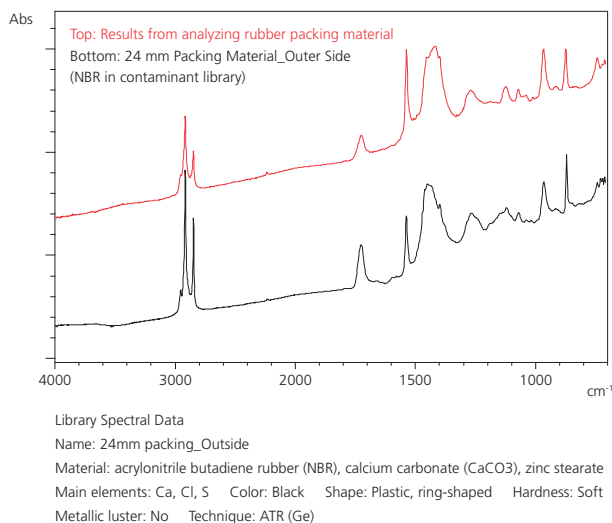


Fig. 1 Results from Analyzing Rubber Packing Material, and NBR Spectrum Held in Contaminant Library

For reference, Fig. 2 shows the results obtained from not using the contaminant library and of comparing the rubber packing material spectrum with the low-additive NBR held in Shimadzu's standard library. Identification is made difficult by the large number of differences between the two waveforms.

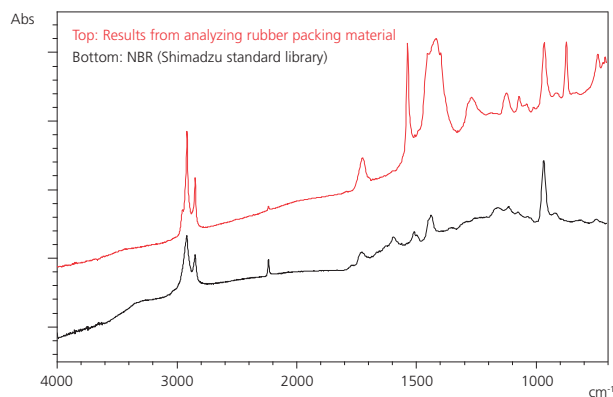


Fig. 2 Results from Analyzing Rubber Packing Material, and NBR Spectrum in Shimadzu's Standard Library

2.2 Rich Variety of Spectra for Food-Related Contaminants

The library contains spectra for actual contaminants collected by food manufacturers. Although many food-related contaminants contain a mixture of multiple constituents, this contaminant library contains many spectra that will almost exactly match the entire waveform of such contaminants.

Fig. 3 shows the results from a spectral search performed after analyzing a bone particle as an example food contaminant. The bone particle contains a mixture of calcium phosphate, proteins and other substances. It matches extremely well with the bone particle spectrum held in the contaminant library. This demonstrates the utility of the contaminant library in identifying samples that contain a mixture of constituents.

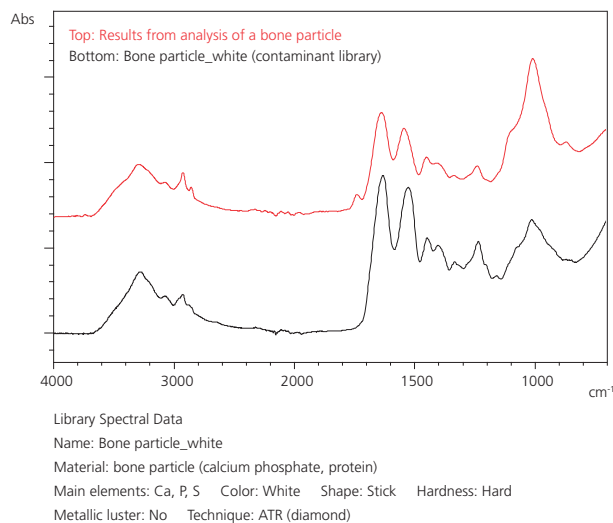


Fig. 3 Results from Analyzing Bone Particle, and Bone Particle Spectrum in Contaminant Library

2.3 Rich Variety of Supplementary Information, such as Shape, Appearance and Constituent Elements

The library also contains a variety of supplementary information on contaminants including that shown below, in addition to infrared spectra.

- The library contains information related to the external appearance of the sample, such as color, shape, hardness, and presence/absence of metal luster. This allows for comparison of the appearance of a contaminant in addition to the spectral form. The library also includes information on constituent elements and an analytical profile of the contaminant obtained using an X-ray fluorescence spectrometer.
- Identifying information provided with the sample, such as the source of the contaminant, is used in the titles of library spectra. This information is important as it shows the circumstances of how the contaminant was collected.
- Also, instead of using chemical constituents in the names of library spectra, contaminants are described using expressions such as "protein mass," "mold," and "small stone" that describe external appearance. General consumers presented with analytical results regarded this terminology as easy to understand.

3. Points to Note Regarding the "Contaminant Library"

Although the mentioned features of the contaminant library are well-received, there are also some issues and points to note regarding the library.

The first of these is with regard to the data held in the library. Since the "Contaminant Library" is a kind of private library of contaminants, the contaminants held within it accurately reflect the materials used in various fields and industries. A forthcoming challenge will be to expand the contents of the library by gathering contaminants from an even wider range of fields and industries.

The second issue is that even when a spectral search produces results that match almost the entire contaminant waveform, analysts are still encouraged to check for characteristic peaks of the identified material. The results from analyzing rubber packing material that were shown in Fig. 1 are again shown in Fig. 4. Detection of the characteristic peaks of NBR, a peak in the region of $2,235\text{ cm}^{-1}$ derived from the $\text{C}\equiv\text{N}$ group in acrylonitrile, and a peak in the region of 966 cm^{-1} derived from the $\text{C}=\text{C}$ -H group in butadiene, has been verified.

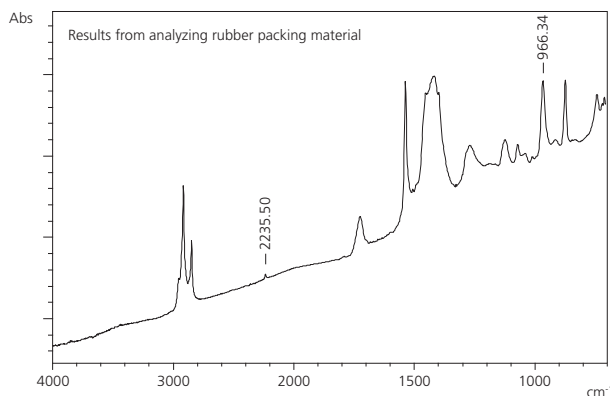


Fig. 4 Verifying the Characteristic Peaks of NBR

The third issue is that when contaminants are discovered in foods and related products, each one is often a mixture of protein, sugar, inorganic material and fat, and these contaminants often exhibit spectral forms that closely resemble one another. Fig. 5 compares the bone particle spectrum from the contaminant library with other materials that give a similar spectral form. It is not easy to discern these differences based solely on the spectral form.

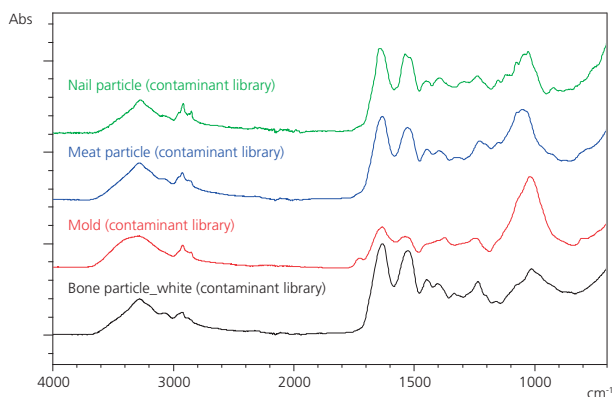


Fig. 5 Spectra Resembling the Bone Particle Spectrum

It hardly bears repeating that making observations of the external appearance of a contaminant and investigating the circumstances of discovery are important parts in an analysis of contaminants. Analysts are also recommended to use an X-ray fluorescence spectrometer or similar instrument to simultaneously verify which elements are contained in the contaminant. As one example, calcium phosphate is an inorganic compound and constituent of bone. In order to verify the presence of calcium phosphate and identify a contaminant as a bone particle, analysts are recommended to verify detection of phosphorous and calcium using an X-ray fluorescence spectrometer.

4. Summary

Contaminants are often a mixture of multiple constituents and exhibit a spectral form that does not match up exactly with normal reference libraries that contain data on single constituents. This makes contaminant identification a difficult task. Using the contaminant library described in this article will provide a number of almost exact matches of the entire spectral waveform of these contaminants. We hope you will consider using this spectral library that has been especially developed to assist with contaminant analysis.

Q&A

Constituents in solution cannot be detected due to strong absorption by water. Is there a solution to this problem?

Water absorbs strongly in the mid-infrared region and creates difficulty in the detection and identification of low-concentration solutes in water. However, detection can be made possible by using a difference spectrum or a concentration technique. This article will briefly describe concentration as a method of analyzing low-concentration constituents. The concentration by evaporation instills a small volume of solution onto an ATR prism and dries the solution before performing analysis. As shown in Fig. 1, water evaporates over time leaving the solute on the prism, thereby concentrating the solution and making the solute easier to identify. (This method is limited to cases in which water evaporates before the solute.)

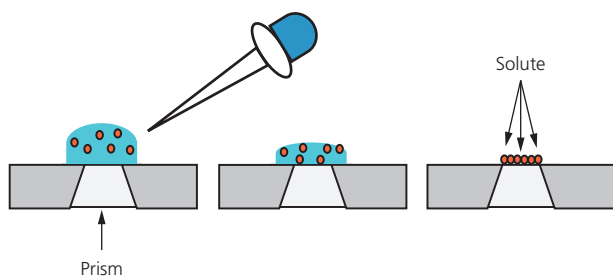
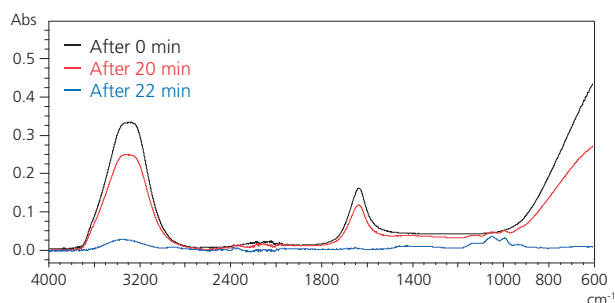
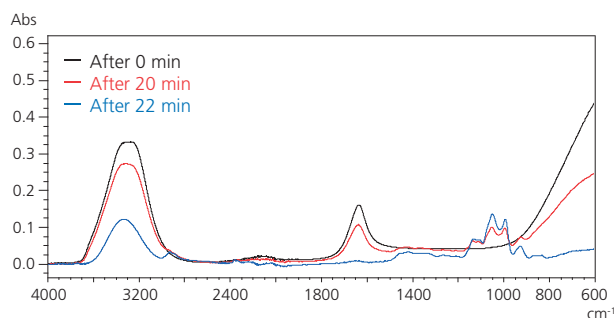


Fig. 1 Schematic Drawing of Concentration by Evaporation Method

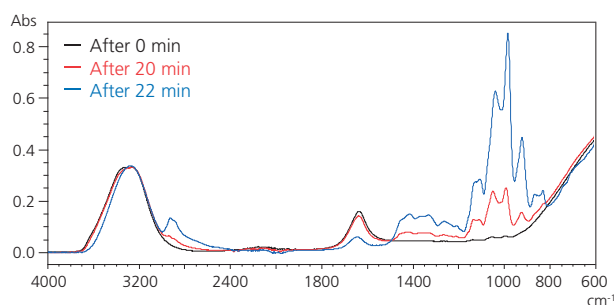
In the following example, 10 μL of an aqueous solution of 0.1 %, 0.5 % or 5 % sucrose was instilled onto a prism and the ATR spectrum was measured. The results obtained from analysis of solutions containing different concentrations of sucrose are shown in Fig. 2 (a), (b) and (c). The results show that over time, there is an increase in the 1,200 to 900 cm^{-1} peak that represents the C-O group characteristic of sucrose, and a decrease in peaks characteristic of water (3,800 to 2,800 cm^{-1} , 1,800 to 1,500 cm^{-1} , 1,000 cm^{-1} and below). When using the method of concentrating the solute by evaporation, an absorption spectrum for sucrose was obtained even at a concentration of 0.1 % sucrose in water. This method therefore enables the detection of low-concentration constituents. Although this example was performed using single-reflection ATR measurements, a multi-reflection ATR prism and a greater volume of instilled solution would allow for analysis of even lower concentration analytes in solution. See Application News No. A551 for details of analytical conditions, etc.



(a) 0.1 % Sucrose Solution in Water



(b) 0.5 % Sucrose Solution in Water



(c) 5 % Sucrose Solution in Water

Fig. 2 Results from Analysis of Sucrose Solution by Concentration by Evaporation Method



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IRSpirit

Ready to Run



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