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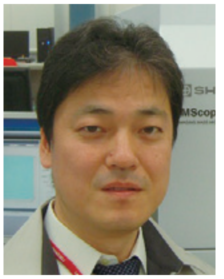
FTIR TALK LETTER

Vol. 30

In Celebration of FTIR TALK LETTER Vol. 30



General Manager, Global Application Development Center
Masaru Furuta

This is the 30th volume of FTIR TALK LETTER, which was first published in 2003. We are extremely grateful to our readers whose support drives us to continue publishing this.

From the very first volume, FTIR TALK LETTER has always consisted of articles describing examples of using FTIR by researchers and analysts, explanations of FTIR terminology by engineers at Shimadzu, and descriptions of related products. We look forward to continue reporting on the usefulness of FTIR by describing applications in various fields.
In explanatory articles, we try to explain basic terminology that even inexperienced readers can feel comfortable reading and we try to answer questions in routine operation.
In articles that introduce products, we have described the features of new products when they are released. Recent products released by Shimadzu include the AIM-9000 automatic failure analysis system (infrared microscope), released in May 2016, and the IRSpirit compact FTIR system, released in October 2017.

In particular, as FTIR analysis has become more widely used for general-purpose applications, compact systems have become especially popular. Shimadzu released the IRSpirit so that we could finally offer customers a compact FTIR model. Due to its small size and reasonable price, a compact FTIR is ideally suited for specific applications.
For example, given that Shimadzu has been compiling a spectral library specifically for contaminant analysis, the compact model was used in combination with a single-reflection ATR accessory and a dedicated macro-program (IR Pilot) to create a dedicated contaminant analyzer that is capable of easily performing all steps from measurement to data analysis and printing. That system was previously featured in an article entitled "Introduction of Shimadzu Compact FTIR Spectrophotometer" (Vol. 29). Such analyzers are anticipated for use in a wide variety of applications. If it is difficult to adequately understand the system based on a written explanation, please feel free to contact a representative at the Global Application Development Center (GADC). GADC exists to serve as a bridge between our products and customers, which is also the purpose of this publication.

It has taken fifteen years to publish the 30th volume. Therefore, I'd like to express my sincere appreciation to the editing team involved in writing and editing the publication and to all the readers that have supported the publication for a long time. We remain committed to providing useful FTIR information in hopes that FTIR TALK LETTER will similarly serve as a vital reference document for FTIR users. Thank you for your ongoing support.

Development Story of IRSpirit (Technology for Miniaturization and Efficiency)

Spectroscopy Business Unit, Analytical & Measuring Instruments Division
Takeshi Maji

1. Prologue

One day when the department was busy preparing for the production of a new product after many years of development, Mr. O, manager of IR Group, Spectroscopy Business Unit asked to meet Mr. K, assistant manager of Mechanical Design Group, Research & Development Department.

"Why not make a FTIR system that is the world's smallest size, while still supporting use of general-purpose optional accessories?" Mr. O asked.

Mr. K knew that markets were demanding smaller analytical instruments for use in laboratories and overseas manufacturers had already released compact FTIR models. However, in order to minimize the size, those models featured unique sample compartment sizes that precluded installing general-purpose optional accessories. Given that the smaller the instrument, the larger the relative size of the sample compartment, the overseas manufacturers seemed to have already indicated that it was impossible to maintain the standard sample compartment size. Just when Mr. K was about to point out that they should focus on launching production of the new product, rather than worry about the compact model, Mr. O pulled out a single sheet of paper from the drawer.

"See! It can fit well!"

He pulled out an A3 paper from the drawer, where all the key components of an FTIR system were crammed together, including the sample compartment, laser, and interferometer. The previous night, Mr. O had cut out drawings of each component and arranged them on the paper.

Certainly, the layout on the A3 paper showed that the system could be made smaller by innovatively reconfiguring the layout with a small semiconductor laser. Furthermore, if the system was combined with user-friendly interactive software developed using expertise at the GADC (Global Application Development Center), the system would offer excellent usability. That ignited a fire under Mr. K's engineering SPIRIT.

"OK. I'll try it." Mr. K answered in a low voice.

That is how development of the IRSpirit started.

The IRSpirit offers the same level of stability, sensitivity, and expandability of higher-end models, but fits them into the size of an A3 sheet of paper.

In addition to IR Pilot navigation software that streamlines (simplifies) the complicated operating steps, the system also includes hardware designed to maximize usability (ease-of-use) and efficiency.

This article describes the technologies used to achieve the smaller size and higher efficiency.

Fig. 1 IRSpirit Compact FTIR

2. Technology for Miniaturization

2.1 Vertical Layout Interferometer

The IRTracer-100 and other higher-end models orient the monochromator horizontally (Fig. 2).

In contrast, the interferometer optics on the IRSpirit were oriented vertically to leave the sample compartment width unchanged from higher-end models and achieve the smallest footprint in its class. (Figs. 3 and 4.)

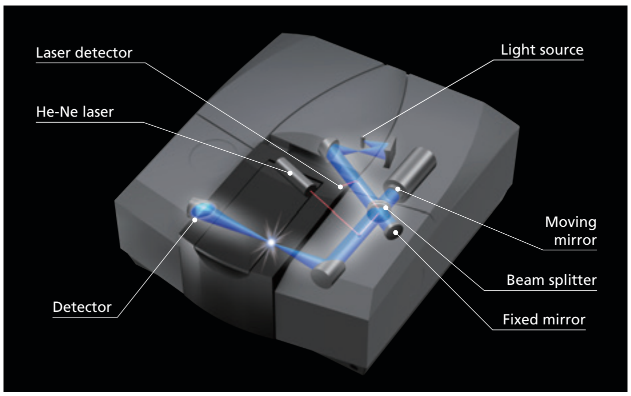


Fig. 2 Layout of the IRTracer-100 Optical System

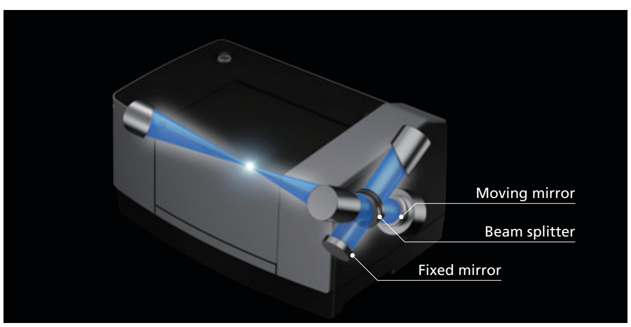


Fig. 3 Layout of the IRSpirit Optical System

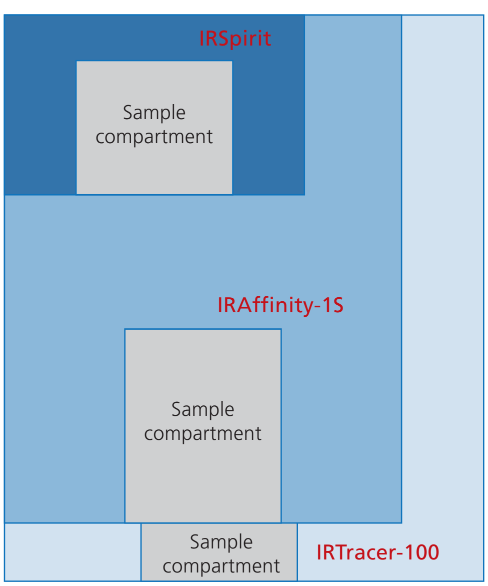


Fig. 4 Size Comparison of FTIR Products

2.2 Change of Laser Optics

Higher-end models use a gas (He-Ne) laser, but the IRSpirit uses a semiconductor laser, which is much smaller.

Higher-end models introduce the light beam by redirecting the path of the light from the externally mounted laser using a mirror in the optical path of the Michelson interferometer, but that resulted in a large instrument (Fig. 5). The IRSpirit achieved a smaller size by eliminating the optics for redirecting the light beam. The laser light from a laser mounted behind the reflection mirror is introduced through a hole in the center of the mirror (Fig. 6). By similarly installing the laser detector behind a reflection mirror with a hole in the center, it prevents leaking laser light directly outside the interferometer and significantly improves instrument safety.

* Pat. No. WO2016166872

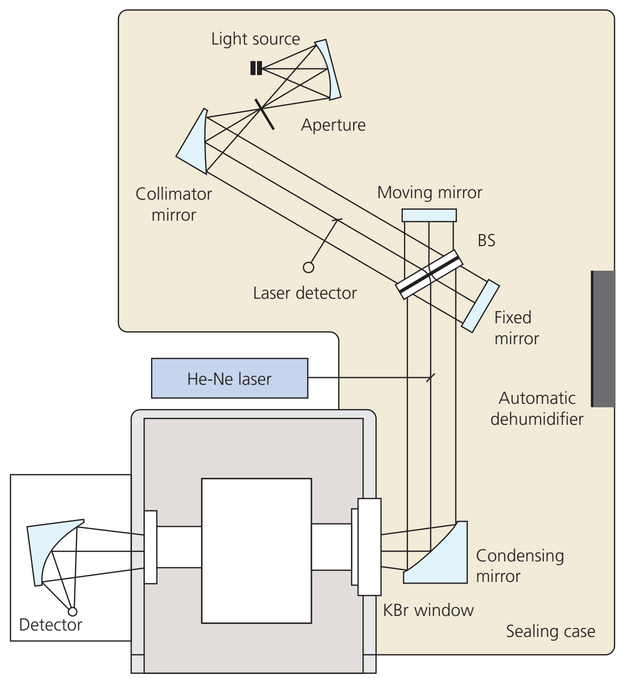


Fig. 5 Layout of the IRTracer-100 Interferometer

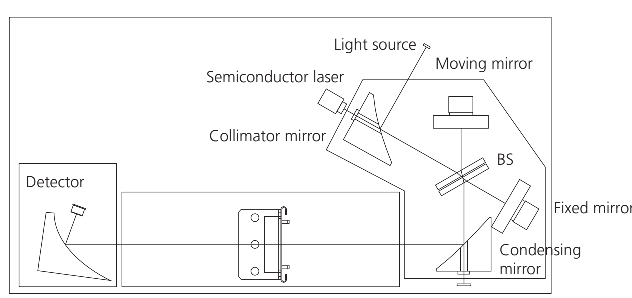


Fig. 6 Layout of the IRSpirit Interferometer

3. Designed for Usability

3.1 Large Sample Compartment

The overall instrument size was reduced to fit on an A3 sheet of paper without compromising the sample compartment size. That means it offers the same expandability as higher-end models by enabling installation of optional accessories available from Shimadzu and other companies.

3.2 Two Orientations Design

To minimize the installation space required for the computer-controlled system, it was designed to enable operation even with the sample compartment oriented toward the right. That means the sample compartment can be operated more easily in especially confined spaces, such as under a fume hood (Fig. 7).

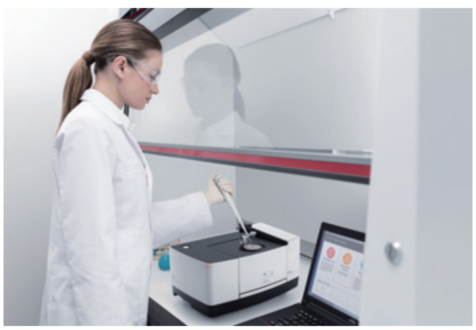


Fig. 7 Using the IRSpirit in a Fume Hood

3.3 Integrated ATR Accessory

The optional QATR-S ATR accessory was designed specifically for the IRSpirit. With the top surface of the prism flush with the top of the instrument, it enables samples larger than the instrument to be measured (Fig. 8). The clamp features a torque-limiter so that samples can be clamped to the prism with a fixed torque. It also provides data with better reproducibility.

Fig. 8 QATR-S Integrated ATR Accessory

3.4 Portable Design

The smaller size decreased the instrument weight to 8.5 kg. In addition, grips are provided at the bottom to facilitate carrying the instrument. However, due to the increased risk of theft, similar to laptop computers, the design includes a hook for attaching a computer security cable or padlock.

4. Design for Intelligence (IR Pilot)

If operations are performed using the newly developed IR Pilot navigation software, operations can be accomplished with only a few mouse clicks, including the printing of reports (Fig. 9). If a measurement program is registered, then it can even be used as a standard operating procedure (SOP).

IR Pilot is knowledge-based software that uses a *wizard* interface based on artificial intelligence (AI). That means users can use the dialogue-based interface to achieve decision making equivalent to an expert operator.

However, given that *wizard* technology has become so commonplace for software installation, few people think of it as AI anymore.

Nevertheless, for the software to enable users to obtain answers that are equivalent to an expert requires that the software include structured expert knowledge. IR Pilot software is only possible because Shimadzu has a vast accumulation of analytical and measurement expertise.

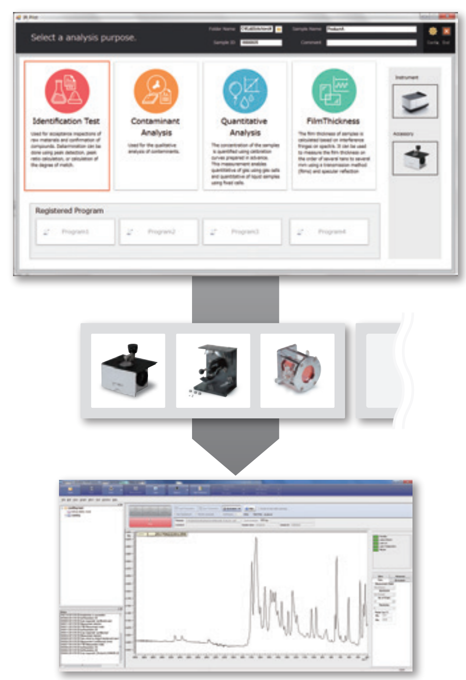


Fig. 9 IR Pilot Navigation Software

5. Summary

This article describes the technologies used to achieve the IRSpirit's smaller size and smarter capabilities.

The small body of the IRSpirit reflects the engineering SPIRIT of the developers.

To satisfy the market needs for a smaller and more efficient model, Shimadzu will continue to develop solutions that combine not only hardware, but also software.

Using EDXIR-Analysis EDX-FTIR Contaminant Finder/Material Inspector

Global Application Development Center, Analytical & Measuring Instruments Division
Haruka Iwamae

Fourier transform infrared (FTIR) spectrophotometers are especially effective for qualitative analysis of organic compounds, but even more accurate qualitative results can be obtained by analyzing the data in combination with element information obtained from an energy dispersive X-ray (EDX) fluorescence spectrometer. The EDXIR-Analysis EDX-FTIR contaminant finder/material inspector analyzes a combination of data acquired by both EDX and FTIR methods. This article describes examples of using EDXIR-Analysis software for contaminant analysis and for satisfying silent change requirements.

1. EDXIR-Analysis EDX-FTIR Contaminant Finder/Material Inspector

Using both element information obtained with an EDX system and compound information obtained with an FTIR system can provide more accurate identification, but extensive experience and expert knowledge of both techniques are essential for identifying substances. Therefore, by incorporating Shimadzu's expertise for analyzing both types of data, we developed specialized software that is able to analyze an integrated combination of both data by simply loading the respective data.

The EDXIR-Analysis process flow chart for analyzing the integrated data is shown in Fig. 1. First, both EDX and FTIR systems are used to measure the same sample and then both types of data are loaded. EDXIR-Analysis classifies samples into three types: organic substances, inorganic substances, or mixtures of organic and inorganic substances, based on the X-ray scattering intensity in the X-ray fluorescence profile. Then the integrated data is analyzed by comparing the data to library data using a unique algorithm for each type. Data analysis results are automatically output as a report. Whether or not optimal data analysis results are obtained depends on whether or not the library includes a spectrum that is similar to the target sample spectrum, just as it does for typical infrared spectral data analysis. (Refer to "Utilizing User Libraries" in FTIR TALK LETTER Vol. 23, which describes how creating and using a private library can be extremely useful for contaminant analysis.) Therefore, to ensure users can build their own library using EDXIR-Analysis, a single button in the results display window can be used to register sample data loaded for analysis as a new library record (registered library data). By building libraries that are specialized for individual users, the accuracy of library searches can be improved with each use.

The contaminant library included standard with the EDXIR-Analysis software includes 485 sets of data registered from analysis by both EDX and FTIR systems. Unlike commercially sold libraries with data mainly for pure substances, this library includes data for mixtures, such as samples actually acquired as contaminants (provided by public water business entities and food companies), gaskets, and so on, which results in significantly higher search accuracy for contaminants and mixtures. It also includes detailed information about samples, such as photographs, color, shape, hardness, and presence of metallic shine or other information obtained visually from the exterior shape, which can be useful for identifying the source of contaminants.

In addition to the included contaminant library, the software also includes functionality for creating user libraries. Examples of library records are shown in Fig. 2. In addition to both EDX and FTIR data, image and PDF files can be registered by linking them to the data. Registered records can also be searched based on keywords in sample names or comments. In addition to serving as a database for analyzing data from unknown samples, it can be used as a tool for storing analytical data from multiple instruments at the same time.

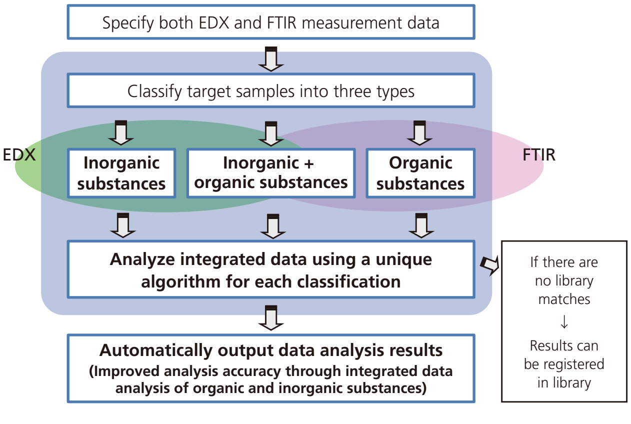


Fig. 1 Flow Chart of Integrated Data Analysis

All Data Is Linked and Stored

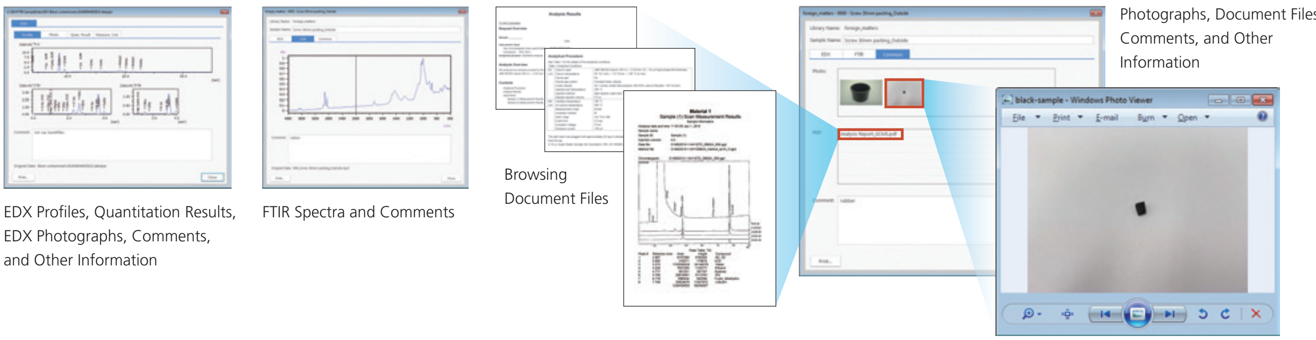


Fig. 2 Examples of Library Records

2. Example of Contaminant Analysis

In this example, a contaminant discovered in food was measured by both EDX and FTIR instruments and then EDXIR-Analysis software was used for integrated data analysis. The instruments and analytical conditions used for the measurements are listed in Table 1. A photograph of the contaminant is shown in Fig. 3. Due to the risk of breaking and scattering the approximately 2 mm × 4 mm hard film-like fragment if it was pressed against an ATR prism during FTIR measurement, the contaminant was held using the EDXIR-Holder, which can be used for either storing or holding contaminants for measurement by either EDX or FTIR. For a description of key features of the EDXIR-Holder, measurement precautions, and so on, refer to Application News No. A537. The analysis procedure is indicated in Table 2.

Table 1 Measuring Conditions

FTIR	
Instrument	: IRSpirit-T (KBr window plate) and QATR-S (wideband diamond crystal)
Resolution	: 4 cm^{-1}
Number of integrations	: 45
Apodization function	: Sqr Triangle
Detector	: DLATGS
EDX	
Instrument	: EDX-8000
X-ray tube target	: Rh
Voltage/current	: 15 kV (C-Sc and S-Ca) and 50 kV (Ti-U) / Auto
Atmosphere	: Vacuum
Analysis area	: 1 mm diameter
Filters	: Without (Ti-U and C-Sc) or #2 (S-Ca)
Integration time	: 60 sec/ch

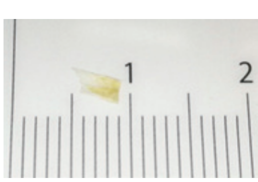


Fig. 3 Appearance of Contaminant

Table 2 Analysis Procedure Using EDXIR-Holder

(1) Affix the contaminant to the adhesive film in the EDXIR-Holder (Fig. 4-1) and perform the FTIR measurement (single-reflection ATR method) with the holder left open (Fig. 4-2).

Fig. 4-1 Attach Contaminants to EDXIR-Holder

Fig. 4-2 Performing the FTIR Measurement

(2) Close the EDXIR-Holder and perform the EDX measurement (Fig. 4-3).

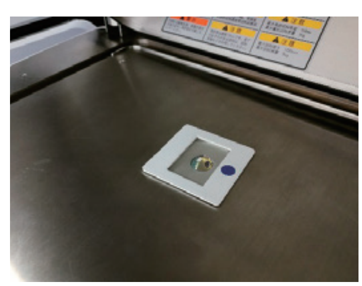


Fig. 4-3 EDXIR-Holder Placed in EDX Sample Compartment

(3) Use EDXIR-Analysis to load both EDX and FTIR data.

Data analysis results displayed in EDXIR-Analysis are shown in Fig. 5. Library records with a high similarity with respect to the sample data are listed at the bottom of the window, with detailed EDX and FTIR data displayed on separate tab pages. On the respective tab pages, X-ray fluorescence profiles or infrared spectra from the sample data and library records in the hit list can be overlaid for comparison. The hit list displays similarity between 0 and 1, where the higher the value, the higher the similarity between data.

Top hits in the hit list from integrated data analysis are shown in Table 3.

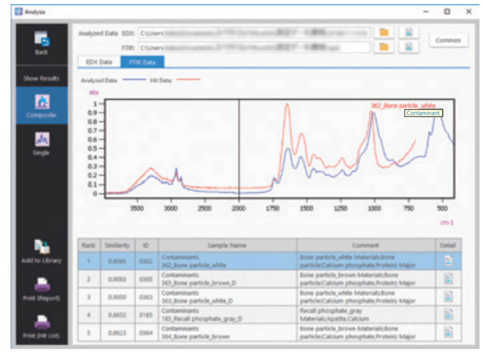


Fig. 5 Integrated Data Analysis Results Window (with [FTIR Data] tab page shown)

Table 3 Hit List from Integrated Data Analysis Results

Rank	Similarity	ID	Sample Name	Comment
1	0.9095	0362	Contaminants 362_Bone particle_white	Bone particle_white Materials;Bone particle(Calcium phosphate,Protein) Major elements;Ca,P,S Color;White Shape;Stick Hardness;Hard Metallic luster;No Technique;ATR(Ge)
2	0.9083	0365	Contaminants 365_Bone particle_brown_D	Bone particle_brown Materials;Bone particle(Calcium phosphate,Protein) Major elements;Ca,P,Mg Color;Brown Shape;Stick Hardness;Hard Metallic luster;No Technique;ATR(Diamond)
3	0.9000	0363	Contaminants 363_Bone particle_white_D	Bone particle_white Materials;Bone particle(Calcium phosphate,Protein) Major elements;Ca,P,S Color;White Shape;Stick Hardness;Hard Metallic luster;No Technique;ATR(Diamond)
4	0.8652	0185	Contaminants 185_Recall phosphate_gray_D	Recall phosphate_gray Materials;Apatite,Calcium phosphate(Ca5(PO4)3) Major elements;Ca,P,Na,Si,Al,Fe Color;Gray Shape;Sand/Cluster Hardness;Soft Metallic luster;No Technique;ATR(Diamond)
5	0.8623	0364	Contaminants 364_Bone particle_brown	Bone particle_brown Materials;Bone particle(Calcium phosphate,Protein) Major elements;Ca,P,Mg Color;Brown Shape;Stick Hardness;Hard Metallic luster;No Technique;ATR(Ge)

The top three records in the hit list in integrated data analysis results were for bone particle. Overlays of the infrared spectra from the contaminant and the first and second bone particle records in the hit list are shown in Fig. 6. Both overlays indicate general similarity of spectral patterns, but the spectrum for hit list record 1 (362_Bone particle_white) matches the contaminant particularly closely. Given the comments in the library record indicate qualitative analysis results of "bone particle", the peaks at 1650 and 1550 cm^{-1} are presumably from protein and the peak near 1000 cm^{-1} from calcium phosphate. The large difference in intensity between peaks near 3000 cm^{-1} and 1750 cm^{-1} in library spectra for 362_Bone particle_white and 365_Bone particle_brown_D is presumably due to fatty acids. Assuming the contaminant is a bone particle, it is very possible an oily substance is on the surface.

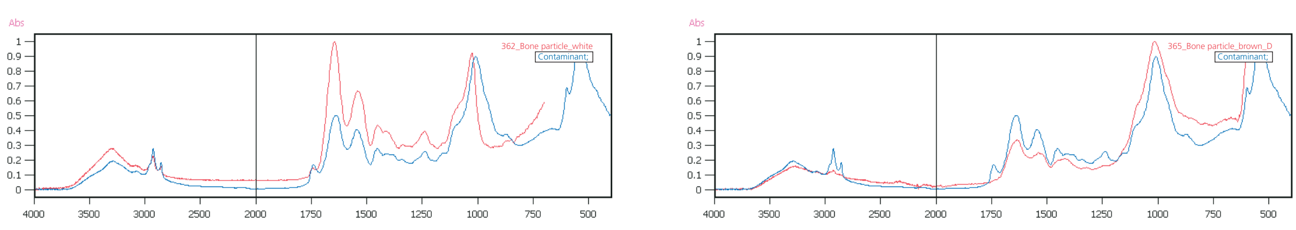


Fig. 6 Comparison of Contaminant and Library Spectra for Bone Particle Left: Bone particle_white; Right: Bone particle_brown

EDX data is confirmed by displaying the other tab page in the data analysis results window. The element content levels in the contaminant and the bone particle included in the hit list are shown in Table 4. It shows that the contaminant contains high levels of Ca, P, Cl, and Mg. That means the elemental composition is similar to the library record for "365_Bone particle_brown_D." The element information obtained by EDX corroborates the presence of calcium phosphate suggested by the FTIR spectra.

Table 4 Element Content in Contaminant and Bone Particle

362 Bone particle_white				365 Bone particle_brown			
Elem	Analyzed	Hit Data	Deviation	Elem	Analyzed	Hit Data	Deviation
Ca	66.2038	74.5442	8.3405	Ca	66.2038	69.8726	3.6689
P	26.5716	21.6673	-4.9043	P	26.5716	26.0432	-0.5283
Cl	3.5272	0.2398	-3.2874	Cl	3.5272	0.4313	-3.0959
Mg	2.4964	0.9059	-1.5905	Mg	2.4964	1.4123	-1.0841
S	0.6759	1.2685	0.5926	S	0.6759	0.3580	-0.3179
K	0.3864	0.6097	0.2233	K	0.3864	0.7684	0.3820
Sr	0.0657	0.2574	0.1917	Sr	0.0657	0.1367	0.0710
Fe	0.0408	0.0441	0.0033	Fe	0.0408	0.1909	0.1501
Zn	0.0223	0.3098	0.2875	Zn	0.0223	0.1798	0.1575
C	0.0100	--------	-0.0100	C	0.0100	--------	-0.0100
Mn	--------	0.0718	0.0718	Al	--------	0.4079	0.4079
Ti	--------	0.0544	0.0544	Mn	--------	0.1722	0.1722
Cu	--------	0.0270	0.0270	Cu	--------	0.0266	0.0266

To confirm the effectiveness of integrated data analysis, let's compare the integrated data analysis results to results obtained by FTIR data analysis alone. Top hits listed from analysis of FTIR data only are shown in Table 5. A comparison of Tables 3 and 5 shows that results from analyzing integrated data and only FTIR data both include bone fragment in the first position of the hit list and the spectral patterns match closely as well. However, the second hit in FTIR-only data analysis results is a cluster of starch, third is mold, and fourth is a plant fragment, which all have an absorption peak near 1000 cm^{-1}, in addition to protein and fatty acid peaks. Figs. 7 and 8 show results from comparing the spectra from the cluster of starch, mold, and contaminant, respectively. Though the peak top and peak shape of peaks near 1000 cm^{-1} differ somewhat, the numerical difference between similarity is not large, which may make it difficult for some analysts, depending on their experience level, to make a determination.

Table 5 Hit List from Data Analysis Results from FTIR Alone

Rank	Similarity	ID	Sample Name	Comment
1	0.8490	0362	Contaminants 362_Bone particle_white	Bone particle_white Materials;Bone particle(Calcium phosphate,Protein) Major elements;Ca,P,S Color;White Shape;Stick Hardness;Hard Metallic luster;No Technique;ATR(Ge)
2	0.8460	0429	Contaminants 429_Cluster of starch_D	Cluster of starch Materials;Starch,Cooking oil(Triacylglycerol),Protein Major elements;Cl,Na Color;Brown Shape;Cluster Hardness;Hard Metallic luster;No Technique;ATR(Diamond)
3	0.8350	0140	Contaminants 140_Mold_D	Mold Materials;Protein,Silicate Major elements;below 1% Color;Brown Shape;Mold Hardness;Soft Metallic luster;No Technique;ATR(Diamond)
4	0.8340	0375	Contaminants 375_Plant fragment2_D	Plant fragment2 Materials;Plant(Cellulose),Vegetable fat(Triacylglycerol) Major elements;Cl,Na Color;Brown Shape;Fragment Hardness;Soft Metallic luster;No Technique;ATR(Diamond)
5	0.8330	0421	Contaminants 421_Cotton textile_D	Cotton textile Materials;Cotton(Cellulose),Protein,Fatty acid ester Major elements;below 1% Color;White Shape;Fiber Hardness;Soft Metallic luster;No Technique;ATR(Diamond)

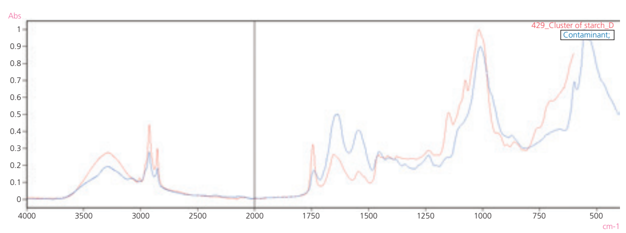


Fig. 7 Comparison of Spectra for Contaminant and Cluster of Starch (Library Data)

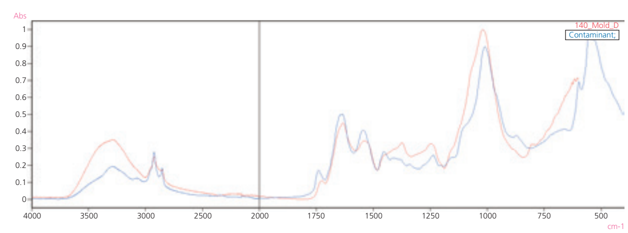


Fig. 8 Comparison of Spectra for Contaminant and Mold (Library Data)

Looking back at the results from analyzing integrated data that includes EDX data, the first three records in the hit list are from bone fragments containing large amounts of Ca and P, which matches the element information for the contaminant in Table 4. Due to confirmation that Ca and P are present in the contaminant, the substance in the library with the closest similarity to the contaminant is bone fragment containing calcium phosphate.

The FTIR results confirmed the presence of protein and fatty acids and also suggests the presence of calcium phosphate. The EDX results confirm the presence of calcium and phosphorus. Therefore, the contaminant is a bone fragment, presumably introduced from a food ingredient.

Consequently, by taking EDX element information into consideration, integrated data analysis using EDXIR-Analysis software can provide more accurate qualitative analysis results, even in cases where it is difficult to choose from multiple candidates determined from FTIR data alone.

3. Example of Satisfying Silent Change Requirements - Using the Data Comparison Function -

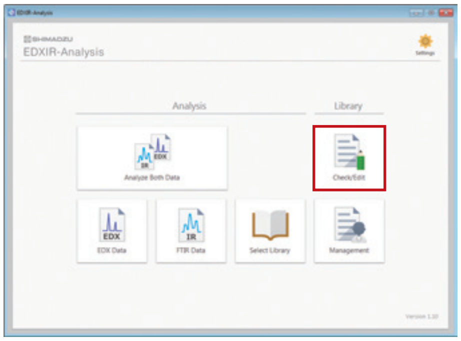
Changes made to ingredients without notifying the trading partners, such as for cost-reduction reasons, are referred to as "silent changes." Silent changes are a problem for society, because using raw materials that do not satisfy specified standards not only prevents ensuring proper product quality, but in some cases can even cause accidents. To ensure high-quality products, it is essential to properly control the safety and quality of raw materials.

Therefore, EDXIR-Analysis includes a data comparison function that can be used for inspecting whether or not deliveries of raw materials are consistent with specified standards. The software can numerically quantify how closely inspected items match the standard items, based on either EDX data, FTIR data alone, or both data. This functionality is especially useful for acceptance inspections, random sampling inspections, and preliminary screening.

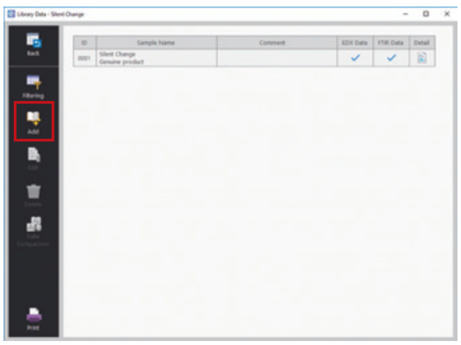
Table 6 shows the procedure for using the data comparison function.

Table 6 Procedure for Using Data Comparison Function

(1) Register data from the standard item in the library.

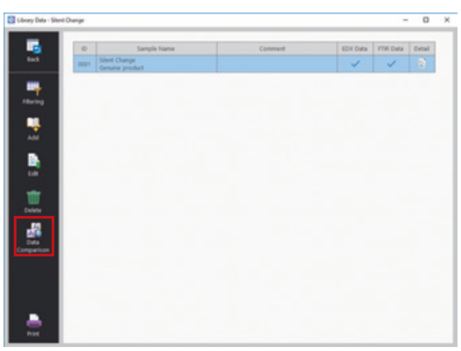


Click the [Library]-[Check/Edit] button and select a library.



Click the [Add] button and register the sample name and comments. Click [Edit] and load EDX or FTIR data. Then click [OK] to register the data.

(2) Compare the data from the inspected and standard items.



With the data from the standard item activated by selecting it in the library data list, click the [Data Comparison] button and load the data for the inspected item. Analyze the EDX or FTIR data alone or perform integrated data analysis of both data.

(3) A data comparison window is displayed that shows similarity calculation results and a comparison of data.

Similarity is calculated for integrated data, EDX data, and FTIR data. Just as in the integrated analysis window, EDX and FTIR data can be confirmed respectively on [EDX Data] and [FTIR Data] tab pages in the data comparison window.

The following describes two examples of comparing data using the data comparison function.

• Plastic Material Containing a Hazardous Substance

The function was used to compare data from a standard polyvinyl chloride (PVC) item and a corresponding inspected PVC item. The results are shown in Figs. 9 and 10. The similarity was 0.8332 based on EDX data alone, 0.8680 based on FTIR data alone, and 0.8506 based on integrated data. From the separate EDX and FTIR data, the X-ray fluorescence profile indicated that the inspected item contained lead (Pb) and the infrared spectrum indicated that it contained a non-PVC substance (marked with ★ symbol) that was not detected in the standard product. Accordingly, it is surmised that the inspected item contains components that are different than those in the standard product.

Fig. 9 Data Comparison Results and X-Ray Fluorescence Profiles

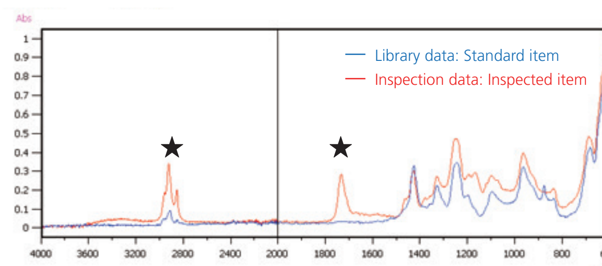


Fig. 10 Comparison of Infrared Spectra

• Difference in Element Content in Plastic Material

Using an ERM-EC680/681 standard polyethylene sample, compliant with the Restriction on Hazardous Substances (RoHS) Directive in Europe, as the standard item, data from an inspected item was compared to data from the standard item. EDX was used for qualitative and quantitative analysis and FTIR to measure the items by the single-reflection ATR method. Respective measuring conditions are shown in Table 7 and results in Figs. 11 and 12.
The X-ray fluorescence profile and infrared spectra overlays for the standard and inspected items showed no differences in the type of principal components contained, but they contained different amounts of S, Cl, Cr, Zn, Br, Cd, Sn, and Sb elements.

Table 7 Instruments and Analytical Conditions

EDX		
Instrument	:	EDX-8000
X-ray tube target	:	Rh
Voltage/current	:	15 kV (C-Sc) and 50 kV (Ti-U) / Auto
Atmosphere	:	Air
Analysis area	:	10 mm diameter
Filters	:	Without (Ti-U and C-Sc), #1 (Rh-Cd), #2 (S-Ca), #3 (Cr-Fe), or #4 (Zn-As and Pb)
Integration time	:	30 sec (without primary filter) 60 sec (with primary filter)
FTIR		
Instrument	:	IRAffinity-1S MIRacle10 (Diamond/ZnSe crystal)
Resolution	:	4 cm^{-1}
Number of integrations	:	40
Apodization function	:	Happ-Genzel
Detector	:	DLATGS

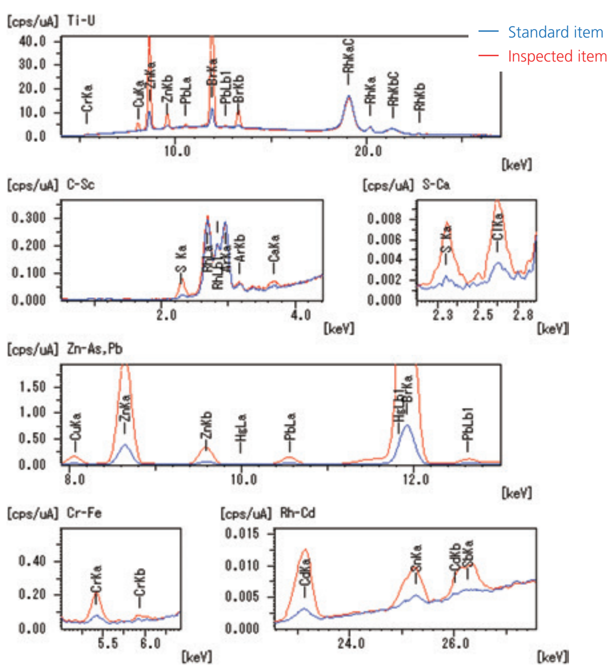


Fig. 11 Comparison of X-Ray Fluorescence Qualitative Analysis Profiles

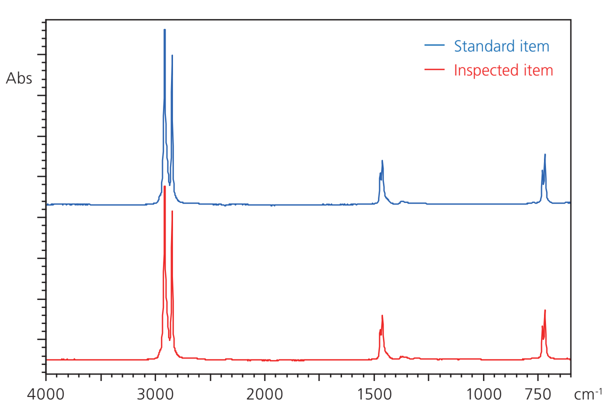


Fig. 12 Comparison of Infrared Spectra

The EDXIR-Analysis data comparison function was used to determine the similarity between two standard items and between a standard and inspected item. Then the reproducibility determined from those scores was used to verify the significant difference between the items. Those results are shown in Table 8. EDX results showed a significant difference, but the FTIR results did not show a significant difference, which corroborated the finding that the inspected item contains different element quantities than the standard item. In this way, differences in samples can be determined more easily by numerically quantifying results, rather than only checking results visually.

Table 8 Similarity Calculation Results

	EDX		FTIR	
n	Standard item	Inspected item	Standard item	Inspected item
1	0.9969	0.9616	0.9790	0.9830
2	0.9948	0.9613	0.9800	0.9800
3	0.9962	0.9613	0.9830	0.9810
4	0.9966	0.9612	0.9820	0.9830
5	0.9960	0.9613	0.9860	0.9790
Mean Value	0.9961	0.9613	0.9820	0.9812
Std. Dev.	0.0008	0.0002	0.0027	0.0018
Significant Difference*	Yes		No	

* Based on t-test (5 % significance level)
Calculated using commercial spreadsheet software

4. Summary

This article describes examples of using EDXIR-Analysis software for contaminant analysis and for satisfying silent change requirements. Considering element information in addition to the organic compound information obtained from regular infrared spectrometry can help to corroborate information obtained from infrared spectra, to determine differences in the element content of samples with no difference in organic components, or to perform more thorough qualitative analysis, even in cases where it is difficult to make a determination based on infrared spectra alone. Try using the EDXIR-Analysis software to perform integrated data analysis based not only on infrared spectra, but also on X-ray fluorescence profiles as well.

References:

Application News

No.A522A Contaminant Analysis Using EDXIR-Analysis Software for Combined EDX-FTIR Analysis

No.A527 Quantifying "Silent Change" Using EDXIR-Analysis Software: EDX-FTIR Contaminant Finder/Material Inspector

No.A537 Introducing the EDXIR-Holder: Sample Holder/Stocker for Contaminant Measurement

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