SHIMADZU
Excellence in Science

Application News

No. A567

Spectrophotometric Analysis

Combined Analysis of a Contaminant Using a Compact FTIR and EDX

Demands regarding the analysis of contaminants that are mixed in or adhered to products are increasing for food and chemical manufacturers and inspection agencies which are consigned inspections.

This increase in demands has drawn attention to energy dispersive X-ray fluorescence spectrometers (EDX) which are suited to analyzing inorganic elements such as metals and to Fourier transform infrared spectrophotometers (FTIR) which are optimal for the analysis of organic substances such as polymeric compounds. Cases where one sample is analyzed using both instruments are increasing as well. However, data obtained with each instrument requires respective analysis procedures and results are sometimes influenced by the operator's knowledge and experience. It is in light of such situations that Shimadzu developed the EDX-FTIR contaminant finder/material inspector, EDXIR-Analysis™ software. The first in the industry, this software is capable of combining and analyzing data acquired from a Shimadzu EDX and FTIR. Details of the software are introduced in Application News Nos. A522A [1] and A527 [2]. This article introduces an example analysis of a contaminant using the EDX-FTIR combined analysis system shown in Fig. 1.

R. Fuji, T. Nakao

Fig. 1 EDX-FTIR Combined Analysis System

Measurement Sample

A contaminant found in a food production process (Fig. 2) was used as the measurement sample. About 4 mm in size and white on the surface, the sample is hard when handled with tweezers. The sample was measured by fixing it in place using the EDXIR-Holder™ shown in Fig. 3, which is a sample holder/stocker for contaminant measurement and effective for streamlining analysis processes for EDX and FTIR. Details of the EDXIR-Holder are introduced in Application News No. A537 [3].

Fig. 2 Photograph of Contaminant Found in a Food Production Process

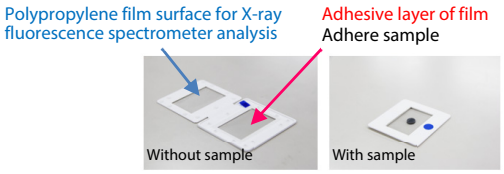


Fig. 3 EDXIR-Holder: Sample Holder/Stocker for Contaminant Measurement

Measurement Using FTIR

IRSpirit™, a compact FTIR, was used for measurement with the QATR™-S single-reflection ATR accessory, which is designed especially for the IRSpirit series, with a diamond prism installed (Fig. 4). Fig. 5 shows the sample set on the instrument and Table 1 lists the measurement conditions that were used. In using the IRSpirit, the dedicated IR Pilot program was used to facilitate measurement. IR Pilot allows operators with minimal FTIR experience to analyze samples by simply selecting the analysis purpose and the accessory. The measured spectrum and the search result from the standards library with the highest similarity are drawn superimposed in Fig. 6. In this case, protein was identified as the best match.

Fig. 4 IRSpirit with QATR-S Single-Reflection ATR Accessory (Diamond prism)

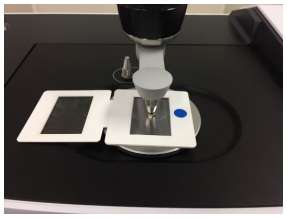


Fig. 5 Sample Set on Instrument

Table 1 Measurement Conditions

Instrument	: IRSpirit-T (KRS-5 window) QATR-S
Resolution	: 4 cm^{-1}
Accumulation Times	: 20
Apodization Function	: SqrTriangle
Detector	: DLATGS

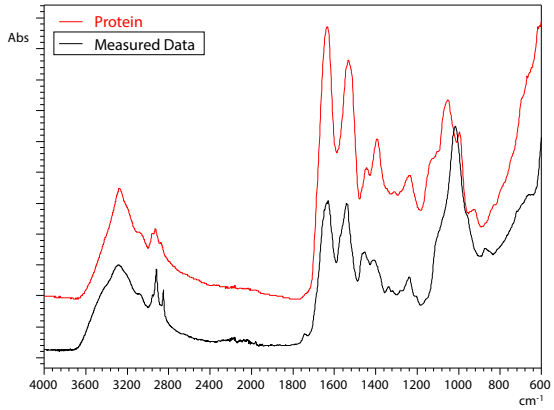


Fig. 6 Infrared Spectra of Measured Data and the Search Result

Measurement Using EDX

Measurement was done using the EDX-7000 energy dispersive X-ray fluorescence spectrometer (Fig. 7) according to the measurement conditions listed in Table 2. The sample was set on the instrument as shown in Fig. 8. For measurement using EDX, the EDXIR-Holder is closed and placed so that the side with polypropylene film is facing the X-ray beam source (bottom). The EDXIR-Holder enables easy setting of samples between EDX and FTIR instruments and contributes to alleviating and improving the efficiency of analysis tasks.

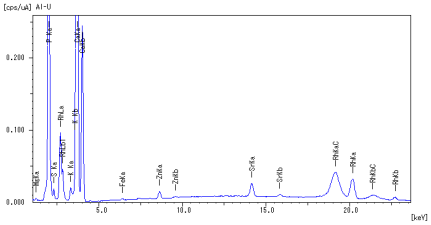
Fig. 7 EDX-7000 Energy Dispersive X-Ray Fluorescence Spectrometer

Fig. 8 Sample Set on Instrument

Table 2 Measurement Conditions

Instrument	: EDX-7000
X-Ray Tube Target	: Rh
Voltage / Current	: 50 kV (Al-U) / Auto
Atmosphere	: Vacuum
Analysis Diameter	: 1 mmϕ
Filter	: None
Integration Time	: 100 s

Fig. 9 shows the qualitative and quantitative analysis results which indicate that $_{20}$Ca and $_{15}$P are the primary elements of the contaminant. Conventionally, the identification of a contaminant requires the analysis of each of the EDX and FTIR measurement results. In this instance however, the EDXIR-Analysis software was used to read and analyze the data acquired from the instruments.



Elements	Ca	P	Mg	K	S	Sr	Zn	Fe
Quantitative Value [wt%]	68.1	28.3	1.8	0.95	0.66	0.09	0.09	0.04

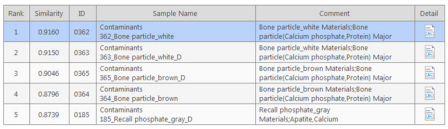
Fig. 9 Qualitative and Quantitative Analysis Results

EDXIR-Analysis, IRTracer, IRAffinity, EDXIR-Holder, IRSpirit and QATR are trademarks of Shimadzu Corporation.

Analysis Using EDXIR-Analysis Software

Analysis was done using the EDXIR-Analysis software. The contaminant library used in analysis was developed by measuring and accumulating data of contaminants provided by water supply organizations and food manufacturers using Shimadzu's EDX and FTIR. Comprising a total of 485 entries, various contaminants such as tap water contaminants and food contaminants are registered.

The hit list shown in Fig. 10 indicates that with a similarity of 0.9160, the most probable match is white bone particle (a mixture of calcium phosphate and protein). Similarity values are within the range from 0 to 1 and larger values indicate that the analyzed data (acquired data) and the hit data (data in the library) are more similar. By comparing the element content and X-ray fluorescence profile of the analyzed data and hit data in Fig. 11 and the infrared spectra in Fig. 12, we can see that the two are highly similar. In addition, images of the sample can be compared as shown in Fig. 13, allowing evaluation of similarity with candidate substances in terms of color, shape, and texture. Based on these various aspects it was concluded that the contaminant is bone.



Rank	Similarity	ID	Sample Name	Comment	Detail
1	0.9160	0362	Contaminants 362_Bone particle_white	Bone particle_white Materials:Bone particle(Calcium phosphate,Protein) Major	
2	0.9150	0363	Contaminants 363_Bone particle_white_D	Bone particle_white Materials:Bone particle(Calcium phosphate,Protein) Major	
3	0.9046	0365	Contaminants 365_Bone particle_brown_D	Bone particle_brown Materials:Bone particle(Calcium phosphate,Protein) Major	
4	0.8796	0364	Contaminants 364_Bone particle_brown	Bone particle_brown Materials:Bone particle(Calcium phosphate,Protein) Major	
5	0.8739	0185	Contaminants 185_Recall phosphate_gray_D	Recall phosphate_gray Materials:Apatite,Calcium	

Fig. 10 Hit List

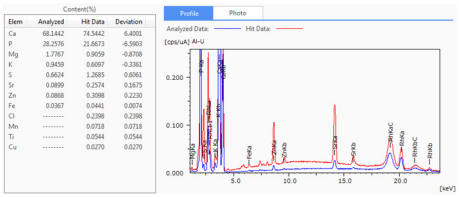


Fig. 11 Element Content and X-Ray Fluorescence Profiles of Analyzed Data and Hit Data

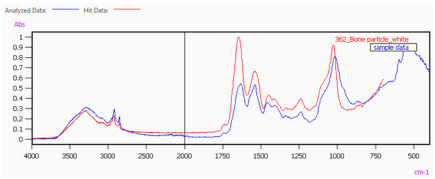
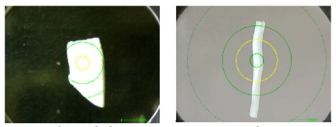


Fig. 12 Infrared Spectra of Analyzed Data and Hit Data



Analyzed data Hit data

Fig. 13 Sample Images of Analyzed Data and Hit Data

The EDXIR-Analysis software enabled easy and swift obtaining of analysis results that combine the inorganic element information acquired using EDX and the organic compound information acquired using FTIR.

References

(1) Application News No. A522A "Contaminant Analysis Using EDXIR-Analysis Software for Combined EDX-FTIR Analysis"
(2) Application News No. A527 "Quantifying "Silent Change" Using EDXIR-Analysis Software: EDX-FTIR Contaminant Finder/Material Inspector"
(3) Application News No. A537 "Introducing the EDXIR-Holder: Sample Holder/Stocker for Contaminant Measurement"

First Edition: Apr. 2018

Shimadzu Corporation
www.shimadzu.com/an/