SHIMADZU
Excellence in Science

Application News

No. A543

Spectrophotometric Analysis

Analysis of Food Contaminants Using KBr Cuttings: KBr Plates for KBr Pellet Formation

The KBr pellet method is a technique mainly used to measure solid samples. This method exploits the plasticity of alkali halides that form a transparent plate when subjected to pressure. While potassium bromide (KBr) is the most common alkali halide used in pellet formation, potassium chloride (KCl) and cesium iodine (CsI) may also be used. Conventionally, pellets were formed by pulverizing KBr and the measurement sample each with an agate mortar, mixing the two to an appropriate concentration, and then applying pressure. However, compared to its crystallized state, crushed KBr readily absorbs moisture and there is also a risk of contamination from the mortar. Furthermore, press-forming work was a burden to analysts and preparing concentrations also took time.

By using KBr Cuttings, the onerous tasks of pulverizing KBr and mixing it with samples using an agate mortar are no longer required. KBr Cuttings are plates of cut KBr crystals. Good quality KBr disks can be produced by simply setting the sample for measurement between two KBr plates, placing the combination into a pelletizer, and applying pressure. When using KBr Cuttings, FTIR measurement is done using the transmittance mode. In this mode, the detector receives a greater amount of light compared to that with the reflectance mode and the ATR method, and therefore features measurement with good sensitivity.

In Application News No. A536, we introduced the procedure for using KBr Cuttings and an example analysis of pharmaceutical identification testing.[*1]
In this article we introduce an example analysis of food contaminants using KBr Cuttings.

R. Fuji

KBr Cuttings Used

Material: KBr
Shape: 3 × 3 × 0.75 mm

Fig. 1 KBr Cuttings

Fig. 1 shows a photo of KBr Cuttings. The shape of KBr Cuttings is either 3 × 3 × 0.75 mm or 5 × 5 × 1 mm.

Analysis of Food Contaminants

Using KBr Cuttings, we measured a black contaminant which was caught in a mesh from filtering inspection, a quality inspection process. Fig. 2 shows the stereo microscope image of the contaminant. The IRTracer-100 Fourier transform infrared spectrophotometer and AIM-9000 infrared microscope, indicated in Fig. 3, were used for measurement. In measurement with an infrared microscope, usage of KBr Cuttings suppresses baseline distortions which occur due to scattering of light at the sample surface as well as interference fringes which may occur when measuring thin samples with a flat and smooth surface. Fig. 4 shows the KBr disk set for measurement. Since the formed KBr disk is the same size as the hole on the sample stage of AIM-9000, the disk can be fixed in place just simply by placing it. Table 1 lists the analysis conditions and Fig. 5 shows the measurement result.

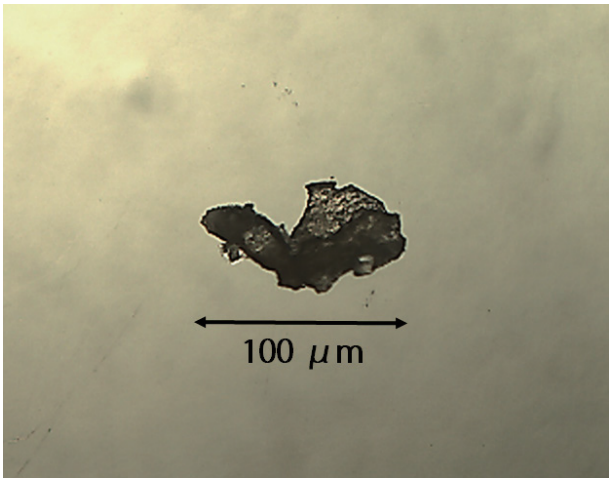

Fig. 2 Stereo Microscope Image of Contaminant

Fig. 3 IRTracer-100 Fourier Transform Infrared Spectrophotometer and AIM-9000 Infrared Microscope

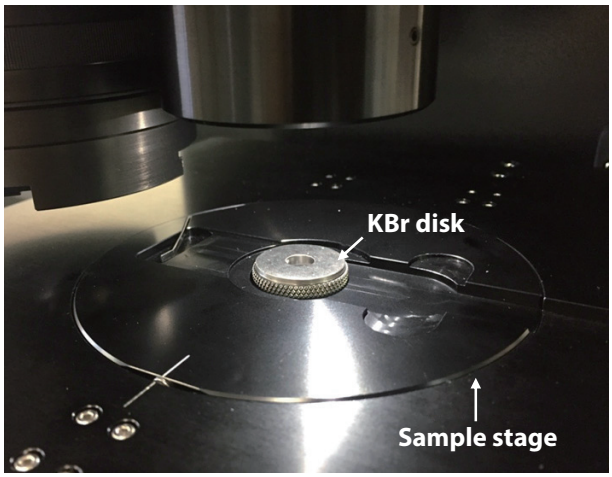

Fig. 4 KBr Disk Set for Measurement

Table 1 Measurement Conditions

Instrument	: IRTracer-100, AIM-9000
Resolution	: 8 cm^{-1}
Accumulation	: 40 times
Apodization function	: Happ-Genzel
Detector	: MCT
Aperture size	: 100 µm × 100 µm

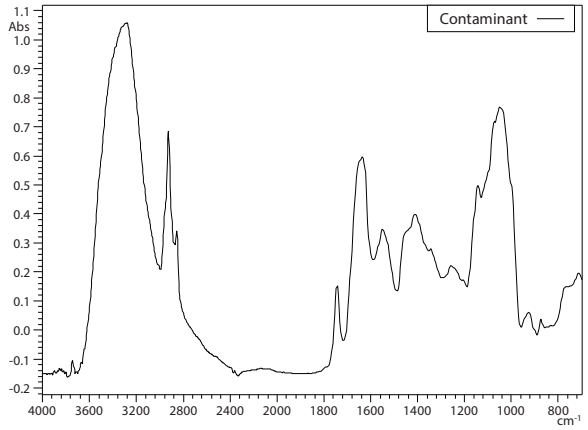

Fig. 5 Measurement Result

The measurement result shown in Fig. 5 indicates a large peak in the vicinity of 3200 cm^{-1} caused by O-H bonds originating from water. Other detected peaks include a peak from C-H bonds near 2800 cm^{-1}, a peak from C=O bonds which are frequently found in foods containing oil near 1750 cm^{-1}, and a peak from amide bonds originating from protein in the range of 1650 to 1550 cm^{-1}.

Analysis of Measurement Result

Contaminant analysis was done using the standard library which contains 12,000 entries. Fig. 6 shows the analysis results.

Based on the library search, the contaminant was found to be a mixture of oil, protein and starch. Since all components are generally included in foods, we can presume that the contaminant is a part of a food.

In analysis of food contaminants, it is often the case that the found contaminant is a part of the food, or food components are attached to the contaminant.
In the latter case, there are times when measurement and analysis are required again after measuring the contaminant and then dissolving the food components such as with water. (The first measurement must be done since the contaminant may also dissolve through pretreatments.) In order to obtain more accurate qualitative results in contaminant analysis, information such as where the contaminant was found and the appearance of the contaminant observed with an optical microscope are also important.

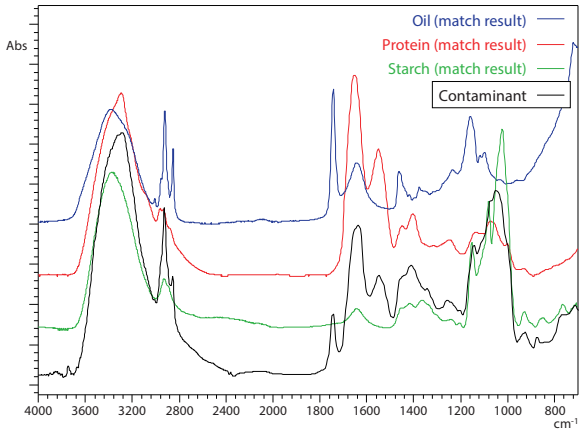

Fig. 6 Analysis Results

Conclusion

We introduced an example analysis of food contaminants using KBr Cuttings. Unlike the conventional KBr pellet method, measurement can be done easily and simply. Also, in addition to measurement with an infrared microscope, KBr disks can be used for the transmittance mode on FTIR instruments. We hope this method will be useful in measurements for contaminant analysis.

References:

*1 Application News No.A536
"Introduction to KBr Cuttings: Convenient KBr Plates for KBr Pellet Formation"

First Edition: Sep. 2017

Shimadzu Corporation
www.shimadzu.com/an/