

Application News

No. A542

Spectrophotometric Analysis

Analysis of Automobile 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 automobile contaminants using KBr Cuttings.

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■ 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 Automobile Contaminants

We measured a black contaminant found on a body production line using KBr Cuttings. Fig. 2 shows the stereo microscope image of the contaminant. The IRAffinity-1S Fourier transform infrared spectrophotometer, indicated in Fig. 3, was used for measurement.

As shown in Fig. 4, the formed KBr disk is fixed to a universal clip holder and placed in the cassette. Table 1 lists the analysis conditions and Fig. 5 shows the measurement result.

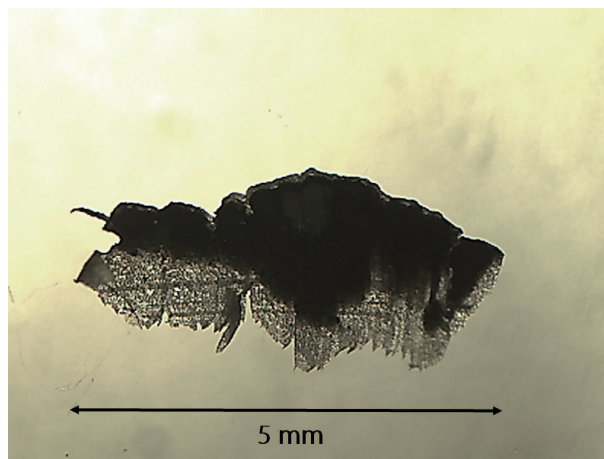


Fig. 2 Stereo Microscope Image of Contaminant

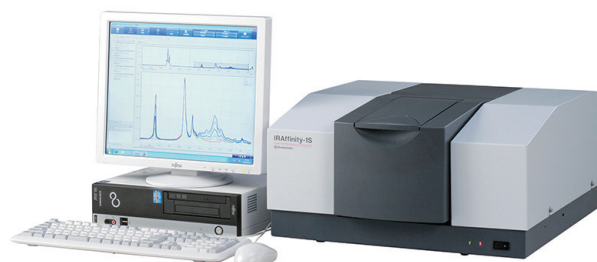


Fig. 3 IRAffinity-1S Fourier Transform Infrared Spectrophotometer

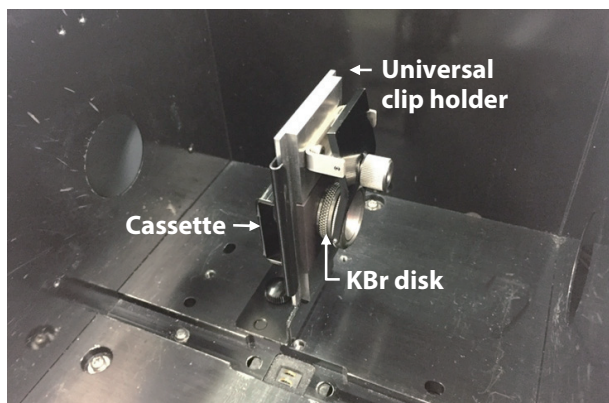


Fig. 4 KBr Disk Set for Measurement

Table 1 Measurement Conditions

Instrument	: IRAffinity-1S
Resolution	: 4 cm ⁻¹
Accumulation	: 40 times
Apodization function	: Happ-Genzel
Detector	: DLATGS

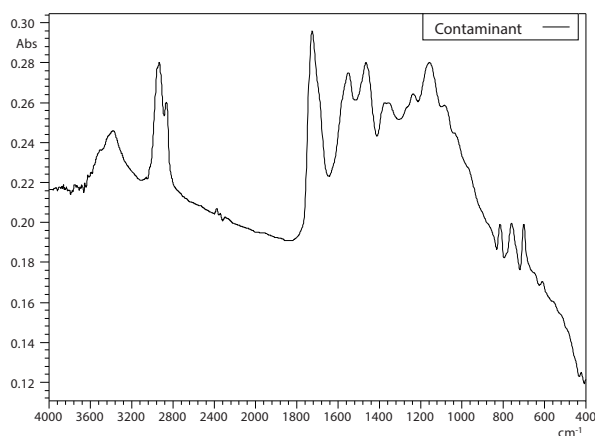


Fig. 5 Measurement Result

The measurement result shown in Fig.5 indicates a large peak in the vicinity of 1750 cm⁻¹ caused by C=O bonds. Other detected peaks include one from C-H bonds near 2800 cm⁻¹ and one from amide bonds near 1500 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 acrylic adhesive and melamine. Since melamine can be considered to be a component of resin paint, we can presume that the contaminant is a fragment of body paint coating.

Measurement using the KBr pellet method conventionally requires pulverizing KBr and the measurement sample each with an agate mortar and mixing the two to an appropriate concentration. However, these processes can be eliminated by using KBr Cuttings. KBr Cuttings allows contaminant analysis using the transmittance mode on an FTIR instrument with procedures as simple as those for the ATR method.

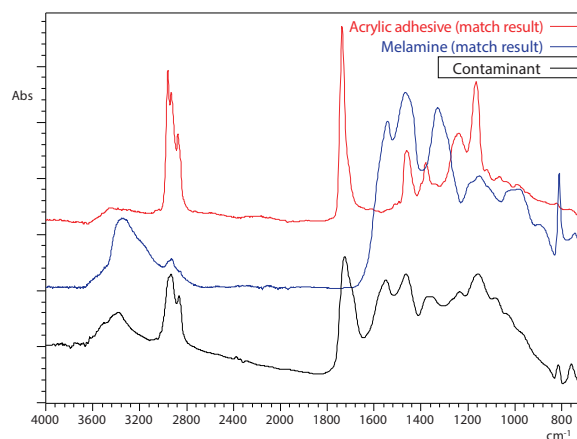


Fig. 6 Analysis Results

■ Conclusion

We introduced an example analysis of automobile contaminants using KBr Cuttings. Unlike the conventional KBr pellet method, measurement can be done easily and simply. In addition, KBr disks can be placed not only on FTIR instruments, but also on the stage of an infrared microscope. 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"