

Application News

No. A536

Spectrophotometric Analysis

Introduction to KBr Cuttings: Convenient 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 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 introduced in this article, 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.

This article introduces the procedure of pellet formation using KBr Cuttings and an analysis example of pharmaceutical identification testing.

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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.

■ Using KBr Cuttings

<Required Tools>

Pelletizer: 7-mm die set

Press: 10-ton hydraulic press

Procedure:

- ① Set one KBr plate onto a 7-mm die pin.
- ② Place the sample on the KBr plate.
- ③ Set the briquetting frame (7-mm collar).
- ④ Set another KBr plate onto the sample.
- ⑤ Set the other 7-mm die pin.
- ⑥ Apply pressure using the hydraulic press.
- ⑦ Release the pressure and retract the press rod.
- ⑧ Removing the 7-mm die pin reveals a finished transparent disk in the briquetting frame.

Fig. 2 shows a photo of each step.

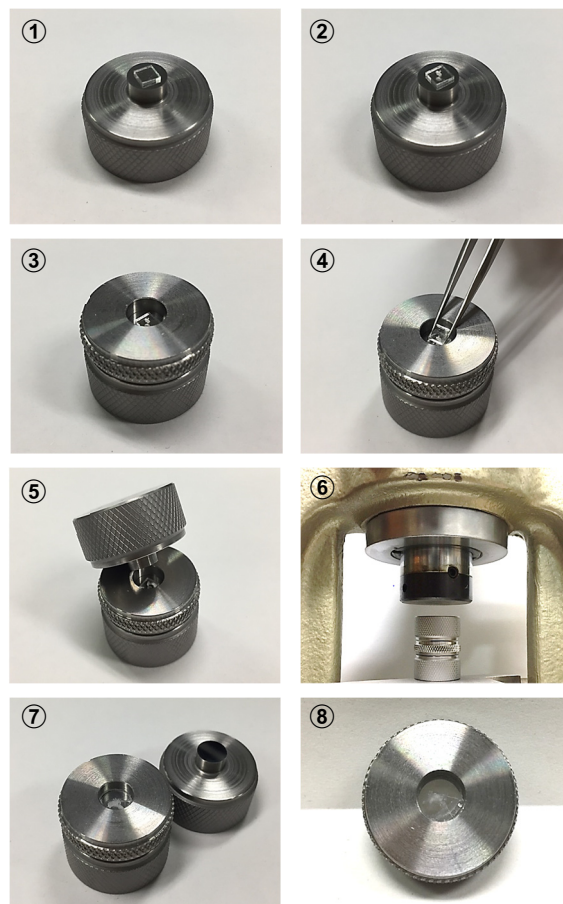


Fig. 2 Using KBr Cuttings

■ Identification Testing of Pharmaceuticals

The Japanese Pharmacopoeia (JP) is an official document that defines the specifications, criteria and standard test methods necessary to properly assure the quality of medicines in Japan. According to the general notices section of this document, "Identification is the test to identify the active ingredient(s) of the drug based upon its specific property". In identification testing that employs FTIR, the absorbance spectrum of a sample and standard are compared, and the identity of the sample and standard are verified to be the same when the same intensity of absorption at the same wavenumbers on both spectra are observed. Although transmission spectroscopy including the potassium bromide (KBr) pellet method, potassium chloride (KCl) pellet method, solution method, paste method, and liquid film method are the common measurement techniques, the 17th edition also allows the use of ATR spectroscopy with respect to some pharmaceuticals, such as montelukast sodium and refined sodium hyaluronate eye drops^{*1}.

The LabSolutions IR software, which controls Shimadzu's FTIR instruments, includes the JP identification test program as standard. Since the testing method for each component is stated in the official monographs of the JP, this program has three functions for handling the wide variety of testing methods. The first function is "peak detection," in which up to 10 peak positions can be specified for checking, and the second function is "spectrum output," which is used to visually compare spectra. The third function is "report creation," which includes the specification of 10 peak positions and up to four points for peak intensity comparison in addition to "spectrum output". For details on each function, refer to Application News No. A454^{*2}.

In this examination we performed an identification test on reserpine, which is used as an antipsychotic and antihypertensive agent. The testing method for reserpine in the JP is stated as "Determine the infrared absorption spectrum of Reserpine, previously dried, as directed in the potassium bromide disk method under Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum previously dried Reserpine Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers"^{*1}. A pellet was formed using KBr Cuttings and an identification test was performed using the "report creation" function in the JP identification test program. Fig.3 shows a photo of the measurement sample and Fig. 4 shows the results of the identification test.

<Measurement Details>

Target component: Reserpine
Test method: Japanese Pharmacopoeia 17th Edition
Measurement method: KBr pellet method

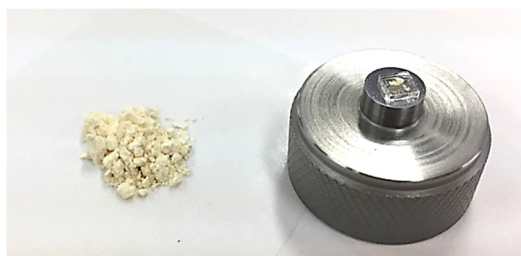


Fig. 3 Measurement Sample

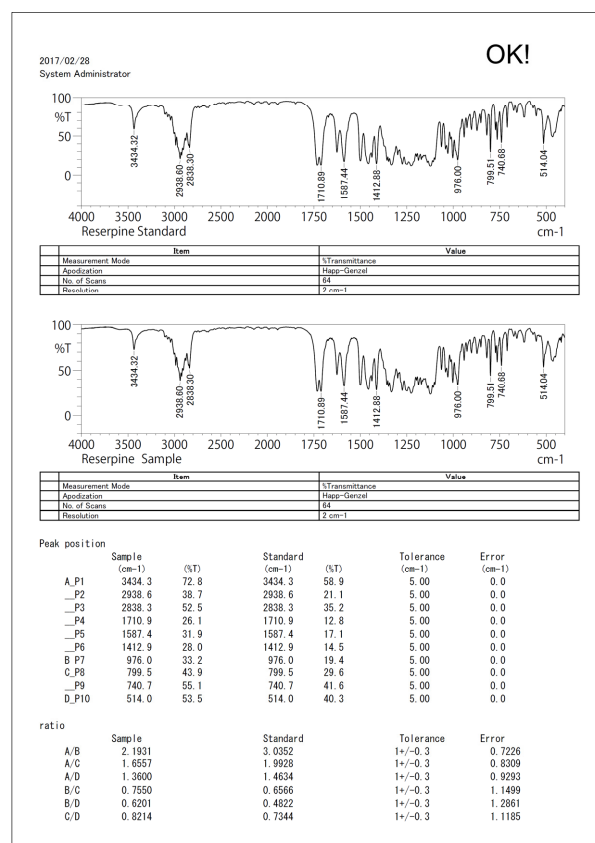


Fig. 4 Identification Test Results

We were able to confirm a pass result as indicated in the upper right of Fig. 4 after performing pass/fail judgment by comparing the standard and reference spectra.

■ Conclusion

In this research, we were able to perform a JP identification test both easily and quickly by utilizing KBr Cuttings. In addition to identification tests, KBr Cuttings can be used as a tool for measuring very small samples with good sensitivity in contaminant analysis and component analysis.

References:

- *1 Ministry of Health, Labour and Welfare of Japan: "Japanese Pharmacopoeia 17th Edition"
- *2 Shimadzu Corporation: Application News No. A454