

Application News

No. A576

Spectrophotometric Analysis

KBr Pellet Formation Using Pixie Mini Hydraulic Press for Pelletizing and Measurement by Compact FTIR

The KBr pellet method is mainly used in measurement of solid samples by the transmission method. This technique utilizes the property that alkali halides possess plasticity and form transparent sheets when subjected to pressure. Production of KBr pellets with high quality and satisfactory transparency is important for obtaining good infrared spectra.

Oil hydraulic presses and mini hand presses are widely used as press machines for pelletizing. Although application of pressure is comparatively simple with hydraulic presses, these machines have a large footprint, and compaction with mini hand presses requires considerable manual labor.

The Pixie portable hydraulic press (manufactured by PIKE Technologies), which features an ergonomic design and has a maximum load of 2.5 t, was developed to solve these problems. Heavy manual labor required with mini hand presses is not necessary, and the device can be used simply with minimal force. Since the Pixie has a small footprint of only W12.7 cm × D19.2 cm, it can also be used in draft chambers. Among other advantages, the pressure gauge is easily visible, and uniform, high quality KBr pellets can be produced by standardized operations by several methods, including those in pharmacopeias, supporting spectrum reproducibility.

This article introduces the results of measurements of KBr pellets produced with the Pixie by Shimadzu's compact FTIR IRSpirit™.

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■ Pixie 2.5 t Portable Hydraulic Press

Figs. 1 and 2 show the appearance and method of applying pressure with the Pixie, respectively. Pressure is applied by first fixing the pelletizing form by tightening the blue knob at the top so that the device will not move, and then turning the hydraulic press knob on the front. Fine pressure adjustment is possible by checking the pressure gauge.



Fig. 1 Pixie 2.5 t Portable Hydraulic Press



Fig. 2 Method of Applying Pressure

■ Reagent Identification Test

We performed an identification test of sodium cholate hydrate using a compact FTIR (IRSpirit) shown in Fig. 3 and the Pharma Report identification test program (Japanese Pharmacopoeia-approved program) included as a standard feature in the FTIR control software LabSolutions™ IR. Table 1 shows the measurement conditions. Here, pellets were made using the KBr Cuttings (3 × 3 × 0.75 mm) shown in Fig. 4 and the 7 mm die set of the pelletizer. For details concerning the forming method, refer to Application News No. A536.¹⁾

The Pharma Report identification test program has three functions to support diverse test methods. The first is "Peak detection," which makes it possible to designate up to 10 peak positions as identification items, the second is "Spectrum output" for visual comparison of spectra, and the third is "Report generation," which makes it possible to designate up to 10 peak positions and up to 4 peak intensity ratios in addition to "Spectrum output." These functions are described in detail in Application News No. A454.²⁾



Fig. 3 Compact FTIR IRSpirit™

Table 1 Measurement Conditions

Instrument	: IRSpirit-T (KRS-5 Window plate)
Resolution	: 2 cm ⁻¹
Accumulation	: 40
Apodization function	: SqrTriangle
Detector	: DLATGS



Fig. 4 KBr Cuttings

The Japanese Pharmacopoeia provides the following identification test method for sodium cholate hydrate: "When measuring this material by the potassium bromide disk method of infrared absorption spectrometry, confirm absorption in the vicinities of the wavenumbers of 3400 cm⁻¹, 2940 cm⁻¹, 1579 cm⁻¹, 1408 cm⁻¹, and 1082 cm⁻¹." ³⁾ Pellets were made by KBr Cuttings, and an identification test was conducted using the "Peak detection" function of the Pharma Report identification test program. Fig. 5 shows the measurement sample, and Fig. 6 shows the result of the identification test.

[Content of Measurement]

Measurement Sample: Sodium Cholate Hydrate

Measurement Method: KBr Pellet Method



Fig. 5 Measurement Sample

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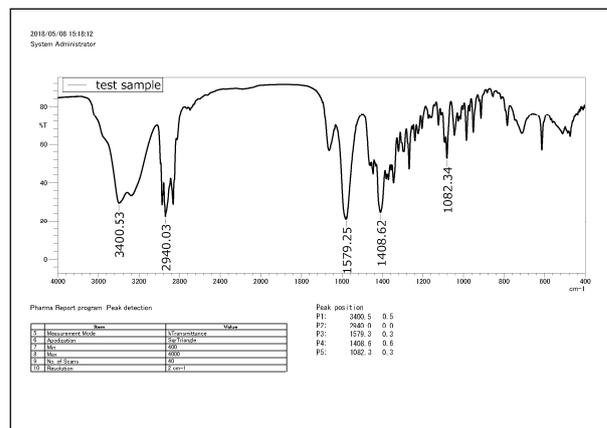


Fig. 6 Results of Identification Test

In the results of the identification test, absorption at wavenumbers of 3400.5 cm⁻¹, 2940.0 cm⁻¹, 1579.3 cm⁻¹, 1408.6 cm⁻¹, and 1082.3 cm⁻¹ was confirmed in the infrared spectrum of the measurement sample obtained by the measurement. These were satisfactory results, as the maximum deviation from the designated peaks was 0.6 cm⁻¹.

Conclusion

It was possible to produce satisfactory KBr pellets simply and with minimal force by using the Pixie portable hydraulic press. The Pixie is a convenient tool when handling large numbers of samples, for example, in daily routine analysis work.

References

- 1) Application News No. A536, "Introduction to KBr Cuttings: Convenient KBr Plates for KBr Pellet Formation"
- 2) Application News No. A454, "Identification Test for D-Galactose and Xylitol Using the Pharma Report Program – Program for Identification Test, Acceptance Check and Pre-Shipment Check –"
- 3) Ministry of Health, Labour and Welfare, "Japanese Pharmacopoeia 17th Ed."