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Application News

No. A545

Spectrophotometric Analysis

Pharmaceutical Excipient Identification Test Using IR Pilot™

The Shimadzu IRSpirit™ is a compact FTIR with a footprint approximately the size of an A3 (11 × 17 in) sheet of paper (Fig. 1). Designed to take up little space, the sample compartment of the IRSpirit is easily accessible whether installed in "landscape" or "portrait" orientation, so that even a narrow opening on a lab bench can accommodate it. The performance it provides is remarkable too, with the highest signal to noise ratio and maximum resolution in its class. These high-level specifications translate into robust results, especially considering its small footprint.

Shimadzu realizes that customers need access to a wide variety of sampling accessories to get the most out of their FTIR. The sampling compartment of the IRSpirit is designed for maximum flexibility, and accommodates existing Shimadzu and third party accessories such as ATR and diffuse reflectance, as well as transmission accessories such as a KBr pellet holder and demountable cells.

Software ease of use is almost as important as its functionality. IR Pilot, an analyzer interface within LabSolutions™ IR, had four common workflows by default: chemical identification, contaminant analysis, quantitative analysis, and film thickness determination. User determined workflows can be created and saved as well. With push-button simplicity, the user selects an analysis, chooses a method and corresponding accessory, picks relevant data manipulation steps, and then starts the measurement. After data collection, a report is created, summarizing data collection parameters and providing interpreted results. This article highlights the features of the IRSpirit, focusing on the Identification Test workflow.

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Fig. 1 Picture of the IRSpirit compact FTIR

Pharmaceutical Excipient Identification Test

Hypromellose acetate succinate is a pharmaceutical excipient used for enteric coatings. It is also used as a carrier for solid dispersions to facilitate miscibility and bioavailability of poorly soluble API. Official compendia such as Japanese Pharmacopoeia (JP) and US National Formulary (NF) describe spectrophotometric methods using FTIR-ATR methods for identification testing. Fig. 2 shows the structural formula of hypromellose acetate succinate.

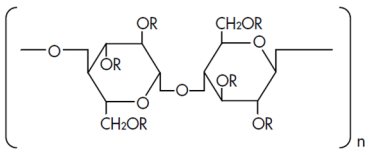

Fig. 2 Structural Formula of Hypromellose Acetate Succinate

The 17th edition of the JP provides a method based on the appearance and position of 5 spectral features, observed at 2840 cm^{-1}, 1737 cm^{-1}, 1371 cm^{-1}, 1231 cm^{-1}, and 1049 cm^{-1} when measured by FTIR-ATR spectroscopy*1.

QATR™-S Dedicated ATR Accessory

The identification measurement was performed with the QATR-S single bounce ATR accessory (Fig. 3), newly designed specifically for the IRSpirit. This accessory mounts in the sample compartment of the IRSpirit, flush on all sides, creating a wide top-sampling surface that can easily accommodate large samples without having to cut them down. Both diamond and germanium crystals are available, and easily user-swappable. The swing clamp mechanism that pushes the sample against the crystal incorporates a torque limiter, preventing damage to the crystal from over-tightening. The QATR-S can be mounted only in the IRSpirit.

Fig. 3 QATR-S ATR accessory mounted in IRSpirit

Identification Test Using IR Pilot

IR Pilot guides the user through sample analysis, a single step at a time, and with minimal user input. For the Hypromellose acetate succinate identification test, the "Identification Test" option in the Main Menu was selected, as shown in Fig. 4. The JP specific method was chosen, which automatically sets the spectral resolution to 2 cm^{-1}. Table 1 show the instrument configuration and settings for this measurement.

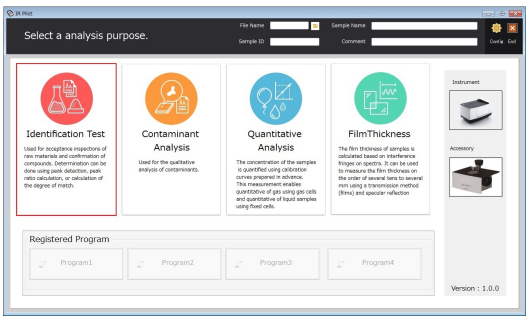

Fig. 4 Main menu of IR Pilot

Table 1 Measurement Conditions

Instrument	: IRSpirit-L (KBr window), QATR-S (Wideband diamond disk)
Resolution	: 2 cm^{-1}
Accumulation	: 45
Apodization	: Sqr Triangle
Detector	: $LiTaO_3$

The measurement method can be selected on the measurement method selection screen (Fig. 5), however, the instrument recognizes QATR-S automatically, and ATR Spectroscopy will be selected. In this case, Fig. 5 screen will not be displayed and the ATR prism selection screen appears (Fig. 6). When the type of the ATR prism is selected, the measurement range will be automatically set. In this case, selection of the diamond crystal sets the useable range to 4000 – 400 cm^{-1}. The IR Pilot workflow leads the user through collection of the spectral background, and then describes placement of the sample to be analyzed on the surface of the QATR-S. By following the prompts, even a first-time user can collect useable data quickly and reliably.

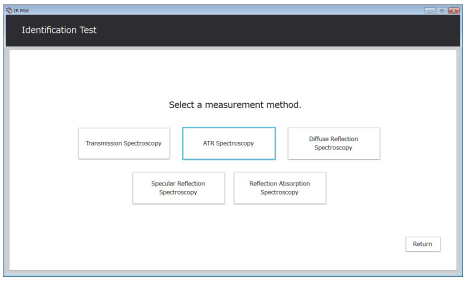

Fig. 5 Selection of Measurement Technique

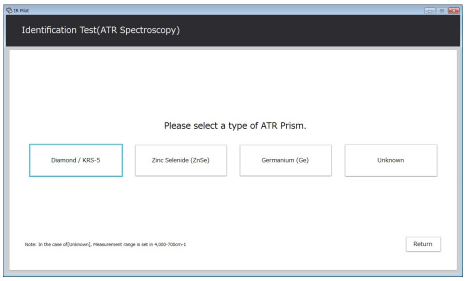

Fig. 6 Specification of ATR Crystal Type

Fig. 7 shows the measurement result. The absorption peaks specified in the JP are observed at 2837 cm^{-1}, 1736 cm^{-1}, 1371 cm^{-1}, 1231 cm^{-1}, and 1048 cm^{-1}.

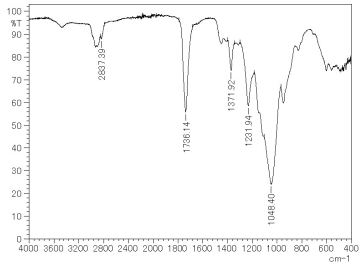

Fig. 7 Spectrum of Hypromellose Acetate Succinate taken with IRSpirit

After data collection, the user is prompted to select post-processing options such as ATR correction, peak picking, peak ratio calculation and purity determination (Fig. 8). In this example, peak pick was chosen.

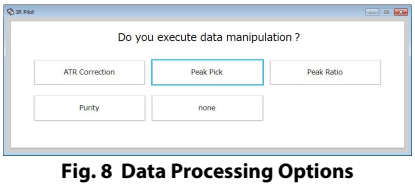

Fig. 8 Data Processing Options

Once data measurement and post-processing are complete, the Report Page is displayed. Fig. 9 shows the format for this, including the measured spectrum and results of any data manipulation.

Once a specific method has been created via the IR Pilot workflow, those settings can be saved by the user (Fig. 10). Both Shimadzu-default as well as User-saved methods can be included as one four options displayed on the main menu. By running methods from the main menu, the number of displayed screens is reduced, saving time and effort.

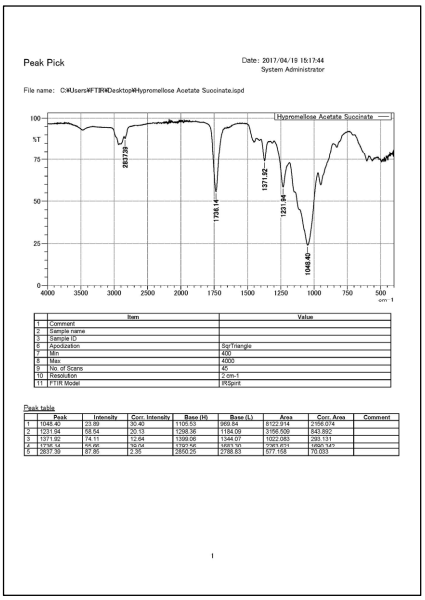

Fig. 9 Example of Default Report

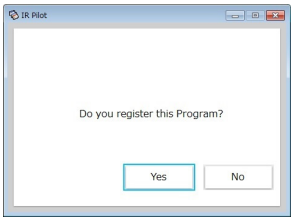

Fig. 10 Register Program to Reuse Method

Conclusion

The identification of hypromellose acetate succinate was performed on the IRSpirit, using IR Pilot to guide the user through the required workflow. In this instance, an Identification Test with Diamond ATR on a powder sample was used to collect data, pick peaks and generate a report. IR Pilot provides a step-by-step interface that enables a non-expert immediate success with sample analysis. IRSpirit in conjunction with IR Pilot offers up to 23 different workflows based on the user purpose, measurement approach, accessory used and sample type. Guidance, flexibility and high quality FTIR data – IRSpirit provides the best combination for customer satisfaction.

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Reference

*1 17th edition of the Japanese Pharmacopoeia published by Ministry of Health, Labor and Welfare

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