

Application News

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Fourier Transform Infrared Spectrophotometer (FTIR)

Identification Testing of Powdered Health Food by IR Pilot™

■ Introduction

The Good Manufacturing Practice (GMP) Guidelines describe requirements that companies must observe across all processes from acceptance of ingredients to packaging and shipping of final products in order to guarantee that products are manufactured appropriately, safely, and with consistent quality. In industries that handle foods and pharmaceutical products, companies are already obligated to observe GMP requirements, and perform tests to confirm that the components and their amounts in foods and drugs are as indicated on the label and the products are free of safety and quality issues. In recent years, a legal obligation to observe GMP in health food and supplement manufacturing processes has also been imposed in Asian countries⁽¹⁾.

This article introduces an example of identification testing of a powdered health food product using the dedicated IR Pilot program, which is an analysis navigation tool of Shimadzu LabSolutions IR software.

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■ IRSpirit™

In Fourier transform infrared spectrophotometry (FTIR), qualitative analysis and quantitative analysis are conducted using the infrared spectrum obtained by irradiating infrared light on the sample. FTIR is the optimum technique for identification testing of samples, as simple and quick measurement of the sample spectrum is possible. The instrument used in the measurements described here was a system consisting of an IRSpirit-T FTIR equipped with a QATR™-S single bounce ATR (attenuated total reflectance) accessory. Fig. 1 shows the external appearance of the system. IRSpirit features a compact and easily portable body with a footprint no larger than a sheet size of A3 paper.



Fig. 1 Appearance of IRSpirit™ + QATR™-S

■ IR Pilot

IR Pilot is a dedicated program installed in the LabSolutions™ IR software used in instrument control and data analysis with the IRSpirit FTIR. IR Pilot is an easy-to-use tool that enables four types of measurement/analysis, including identification testing, contaminant analysis, quantitative analysis, and film thickness measurement, and provides user guidance which ensures that all operations from measurement to analysis and printout of the results are carried out by the correct procedures.

Matching calculation is one type of data processing used in identification testing after sample measurement. The infrared spectra of a standard sample (genuine product) and the target item are compared, and the degree of similarity is calculated from a 2-dimensional plot of the absorbances of the two samples at each wavenumber. The degree of similarity is expressed by a correlation coefficient from 0 to 1, with larger coefficients indicating greater similarity between the two spectra. Thus, a coefficient of 1 indicates an exact match between the two spectra. Management using matching calculations is one effective technique for identification testing and quality control of food ingredients, food additives, and pharmaceutical products. When the spectrum of the standard sample and the threshold value are set and a matching calculation is executed, IR Pilot displays the infrared spectra, score, and pass/fail judgment.

■ Identification Testing of Powdered Health Food

Samples were prepared by intentionally mixing a contaminant (sugar) in a commercially-available powdered health food (main ingredient: milk protein). Samples consisting of (a) 100 % sugar, (b) 30 %, (c) 20 %, (d) 10 %, (e) 5 %, and (f) 0 % mixed sugar, and (g) powdered health food, which was the standard sample, were measured by ATR. Table 1 shows the measurement conditions, Fig. 2 shows the ATR spectra of the samples, and Table 2 shows the matching calculation results with reference to the standard sample, together with the pass/fail judgments.

Table 1 Measurement Conditions

Instrument	: IRSpirit-T (KBr window plate) QATR-S (wideband diamond disk)
Resolution	: 4 cm ⁻¹
Accumulation	: 40 times
Apodization function	: Sqr-Triangle
Detector	: DLATGS

The main peaks originating from sugar in the ATR spectrum of (a) 100 % sugar are in the vicinity of 1,049 cm⁻¹ (C-O stretching vibration), 988 cm⁻¹ (C-O stretching vibration), and 908 cm⁻¹ (ring vibration). Although these peaks can be clearly seen in (b) 30 % mixed sugar, judgment of contamination becomes progressively more difficult as the concentration of the contaminant decreases to (c) 20 %, (d) 10 %, and (e) 5 %. However, if the degree of similarity with reference to (g) standard sample is obtained, judgment is possible not only from the peak shape of the spectra, but also by the numerical value of the degree of similarity, which expresses the difference between the standard sample and the test sample. The degree of similarity of sample (f), which contains no mixed sugar, is 0.99 or more, indicating high similarity in comparison with test samples (b) to (e), which contain various amounts of mixed sugar.

By using IR Pilot, it is possible to obtain the results of the identification test, as shown in Fig. 3, by registering 0.9900 as the threshold value for evaluation of the degree of similarity with the infrared spectrum of the standard sample.

Table 2 Results of Matching Calculations and Pass/Fail Judgments

Test sample	Degree of similarity	Pass/fail judgment *
Milk protein+ sugar 30 % (b)	0.8741	Fail
Milk protein+ sugar 20 % (c)	0.9325	Fail
Milk protein+ sugar 10 % (d)	0.9829	Fail
Milk protein+ sugar 5 % (e)	0.9879	Fail
Milk protein+ sugar 0 % (f)	0.9943	Pass

* Threshold value: 0.9900

Conclusion

Identification tests of health foods and supplements can be conducted simply and quickly by using the matching calculation function of IR Pilot. If the test conditions are registered in advance, pass/fail judgment can be obtained simply by measuring the test sample, enabling efficient routine identification testing.

IR Pilot, IR Spirit, QATR, and LabSolutions are trademarks of Shimadzu Corporation in Japan and/or other countries.

<Reference>

- (1) Website of General Incorporated Association, The Japan Institute for Health Food Standards
<http://www.jihfs.jp/gmpn01.html>

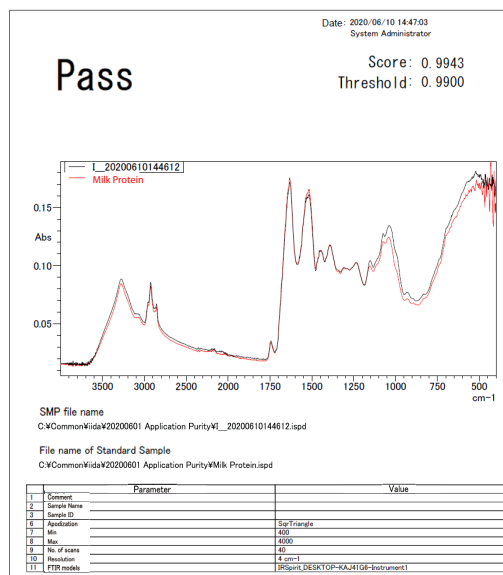


Fig. 3 Results of Identification Test

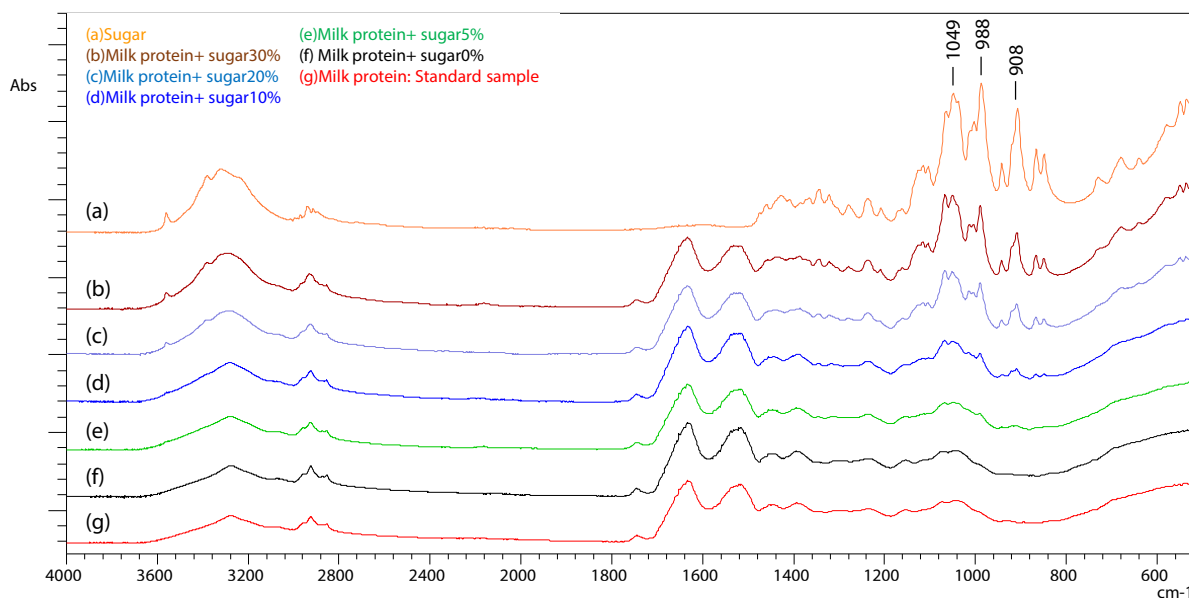


Fig. 2 ATR Spectra of Samples