SHIMADZU
Excellence in Science

Specification Sheet

IRSpirit

Fourier Transform Infrared Spectrophotometer

IRSpirit is a compact Fourier Transform Infrared Spectrophotometer that travels where it's needed.
It gracefully accommodates available bench space and the sample compartment is easily accessible with the system facing forward or sideways.
In addition, IRSpirit is equipped with the widest sample compartment in its class, to easily accommodate both Shimadzu and third-party accessories. IRSpirit also offers the highest S/N ratio (30,000:1*[1]) in its class using the technology inherited from the high-end model.
Furthermore, a newly developed dedicated IR Pilot program*[2] offers 23 standard application programs to simplify analysis for all users.

*1: DLATGS detector with KBr window
*2: Quantitative analysis function cannot be used when using IR Pilot with LabSolutions DB/CS.

IRSpirit Series Hardware

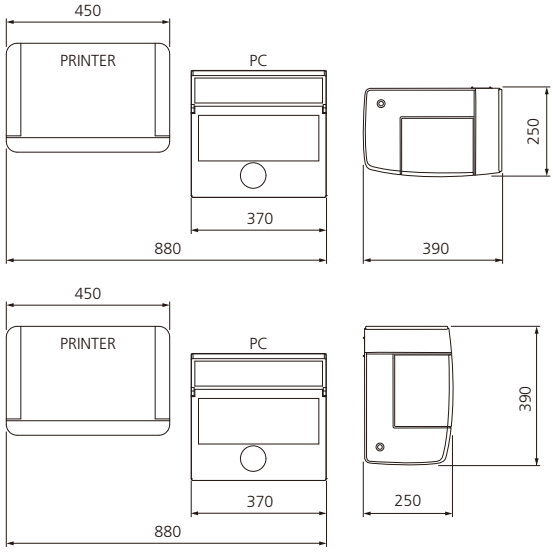
Interferometer	Michelson interferometer (30° incident angle) Equipped with Dynamic Alignment system Sealed interferometer with desiccant
Optical system	Single-beam optics
Beam splitter	Germanium-coated KBr
Light source	High-energy ceramic 3 years guaranteed
Detector	IRSpirit-T model: DLATGS detector with temperature control IRSpirit-L model: $LiTaO_3$ detector
Wavenumber range	7,800 to 350 cm^{-1}
Wavenumber precision	±0.0005cm^{-1} (@2,000cm^{-1})
Wavenumber accuracy	±0.1cm^{-1} (Calculate wavenumber accuracy at wavenumber correction position)
Resolution	0.9, 2, 4, 8, 16 cm^{-1}
S/N ratio	IRSpirit-T model with KBr window: 30,000:1 IRSpirit-T model with KRS-5 window: 23,000:1 IRSpirit-L model with KBr window: 13,000:1 IRSpirit-L model with KRS-5 window: 10,000:1 (peak-to-peak, 4 cm^{-1} resolution, in a neighborhood of 2,100 cm^{-1}, 1-minute accumulation)

Mirror speed	2.8mm/sec
Data sampling	Semiconductor laser with temperature control 6 years guaranteed
Sample compartment	Equipped with automatic accessory recognition mechanism 200(W) × 140(D) × 100(H) mm Center focus
Dimensions	390(W) × 250(D) × 210(H) mm
Weight	8.5kg
Required environment	Operating guaranteed temperature :10 to 35°C Performance guaranteed temperature :15 to 30°C Humidity: 20-70% (KBr window model) 20-90% (KRS-5 window model) with no condensation
Utility	AC 100 to 240V, 50-60 Hz
Power consumption	75 VA (when used), 7 VA (standby)

LabSolutions IR Software

Operating system	Microsoft Windows 7 Professional 32/64-bit edition Microsoft Windows 10 Pro 64-bit edition
Interface	USB 2.0, USB 3.0
Programs	Postrun program Spectrum measurement program Quantitative measurement program Photometric measurement program Time course measurement program (option)
Measurement	Spectrum measurement, continuous measurement, atmospheric correction measurement, simple measurement mode
Hardware monitor	Self-diagnosis function, status monitor
Manipulation functions	Four arithmetic operations, normalization, baseline correction, smoothing, derivative, X-axis range change, data correction, data points thinning, interpolation, wavenumber-wavelength conversion, X adjust, time-temperature conversion, peak pick, point pick, film thickness calculation, data calculation, degree of coincidence, deconvolution, Fourier transform, Kubelka Munk conversion, ATR correction, advanced ATR correction, Kramers Kronig transformation, atmosphere correction, degree of coincidence calculation
Manipulation functions (option)	Peak split, 3D recalculation, spectrum extraction from 3D data
Analysis support programs	Contaminant Analysis, Pharma Report, Food Additives Identification, Purity calculation
Search functions	Spectrum search, peak search, text search, combination search, setting of search conditions, search of user library and commercial library, creation of user library. 2000 types of libraries for analysis of general organic substances, polymers, pharmaceutical products, inorganic substances, food additives, and contaminants.
Quantitative functions	Multi-point calibration curve method CLS quantitative method PLS quantitative method (option) Photometrics Recalculation function for quantitative and photometric results
Printing functions	Report template creation Printing using report templates Easy printing using the ViewPrint function
Validation program	Complies with Chinese, European, US, and Japanese Pharmacopoeias and ASTM
GLP/GMP support	Tree-structured audit trail function Recording of operation logs and data logs (history) Saving by overwriting the same filename is prohibited
Security functions	Coordination with LabSolutions security functions User-group based privilege settings
Macro functions	Dedicated IR Pilot program 23 types of dedicated macro programs for contaminant analysis, identification, quantitation and film thickness calculation are included. Easy macro function • Collective execution of multiple operations by simply arranging operations in the order of the procedure • Execution possible from the desktop Macro platform (option)
Optional software	Time course, PLS Quantitation, Peak split, 3D processing, Macro platform
Optional usable libraries	Contaminant Library for LabSolutions IR, Thermal-damaged plastic library, Saddler library, STJ library, etc.
File formats	Files of JCAMP-DX, ASCII, CSV, IRsolution, HYPER-IR can be loaded and saved.

External Dimensions with Laptop



Unit: mm

SHIMADZU

Shimadzu Corporation

www.shimadzu.com/an/

C103-E114

SHIMADZU
Excellence in Science

Fourier Transform Infrared Spectrophotometer

IRSpirit


SHIMADZU
IRSpirit
FOURIER TRANSFORM INFRARED SPECTROPHOTOMETER

IRSpirit, Ready to Run

Space-Efficient with High Expandability

- Compact FTIR that travels where it's needed.
- For sites with only a narrow space available, samples can be measured with the unit positioned horizontally or vertically.
- With the widest sample compartment in its class, it easily accommodates Shimadzu and third-party accessories.

Dedicated IR Pilot Program Ensures Immediate and Easy System Operation

- IR Pilot includes 23 application programs as standard.
- Includes an identification test program convenient for routine inspections as standard.
- Includes a pass/fail judgment program specialized for contaminant analysis as standard.

High Reliability Ensures the System Can Be Introduced with Confidence

- Stable interferometer performance based on technology inherited from high-end models.
- Designed to endure even high-humidity environments (KRS-5 window is selectable).
- Instrument status monitoring function enables users to understand the instrument status easily.
- Anti-theft and anti-drop keylock can be installed.

Space-Efficient with High Expandability

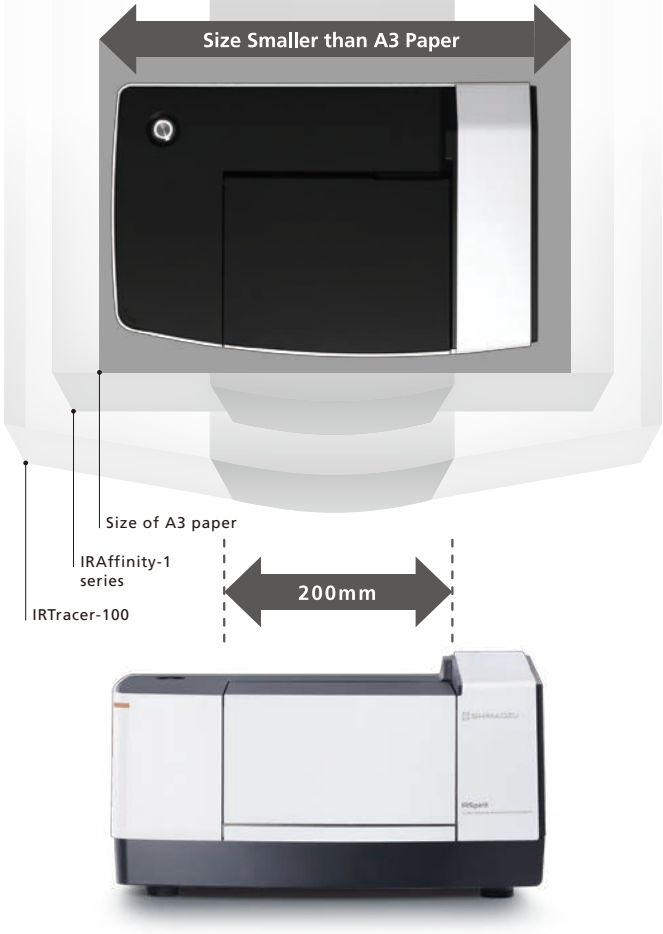
Even Fits in Small Spaces

There is a growing need for systems that can fit in tight spaces, such as for lining up a row of units used for student experiments or to enable measurements in an environment with many different samples and experimental tools laid out for use in chemical synthesis. For sites with only a narrow space available, samples can also be measured with the unit positioned vertically (see diagram below). The start switch is accessible and the humidity indicator is visible from both directions.
The prism and FTIR main unit are the same in height. Therefore, samples can be placed directly on the ATR attachment, which is integrated with the sample compartment. That can eliminate the trouble of having to cut large samples.


HUMOTY

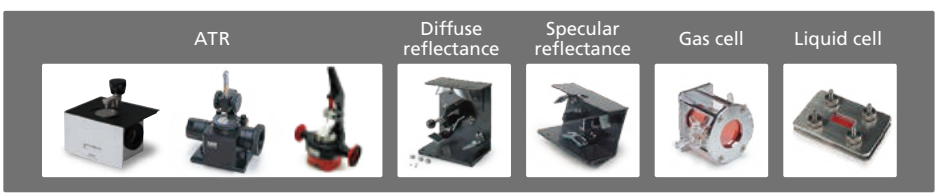
Large Sample Compartment in a Small Body

In spite of a body size smaller than a piece of A3 paper, the sample compartment width is the same as on higher-end models. This makes it compatible with many Shimadzu and 3rd party accessories, allowing it to be used for a wide variety of applications.



Fields \ Applications		Contaminant analysis	Raw materials acceptance inspection	Identification tests	Quantitative analysis	Spectral analysis
Pharmaceuticals and life sciences		○	○	○	○	
Chemicals and petroleum		○	○	○		
Academia				○	○	○
Environmental		○			○	○

○ : Applicable



Dedicated IR Pilot Program Ensures Immediate and Easy System Operation

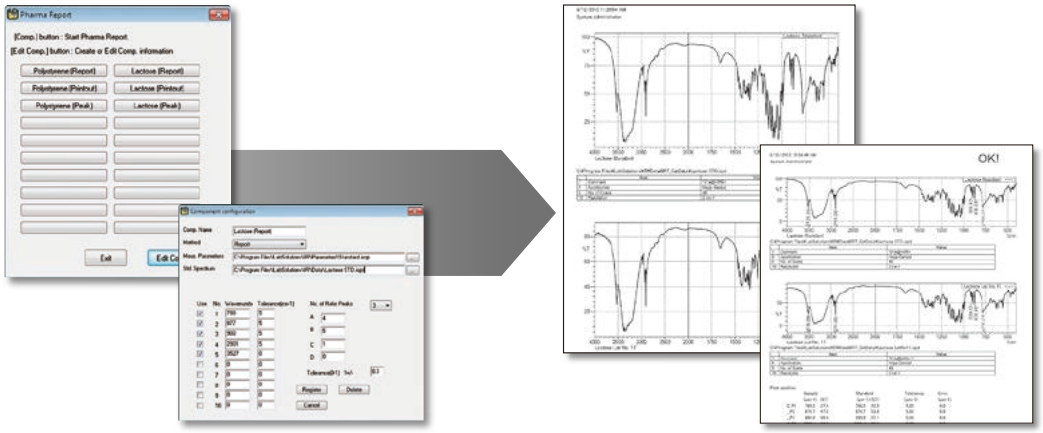
IR Pilot

IR Pilot offers a total of 23 application programs as standard, making it easy for operators with minimal FTIR experience to analyze samples by simply selecting the analysis purpose and accessory. There is no need to set parameters. It enables the measurement of multiple samples with only one click.

* Quantitative analysis function cannot be used when using IR Pilot with LabSolutions DB/CS.

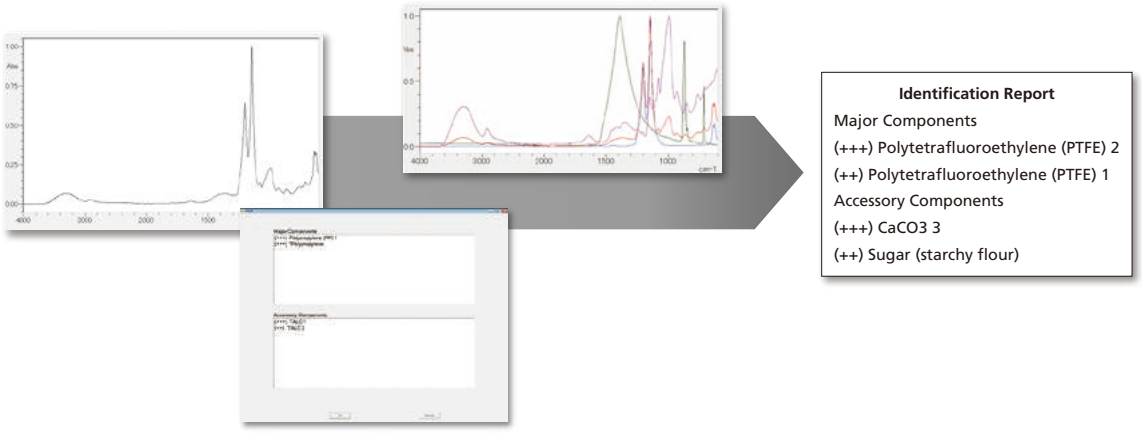
Identification Test Program

This program makes pass/fail judgments for test samples based on verification methods described in Pharmacopoeia and standards specified in each country, such as "Infrared Spectrophotometry" in the Japanese Pharmacopoeia and Japan's Specifications and Standards for Food Additives. In addition to identification tests for pharmaceutical and food product identification tests, the program can also be used for acceptance and pre-shipment inspections.
The program calculates the difference between peak wavenumbers from standard and test samples and the difference between the peak intensity ratios and then prints a report of pass/fail judgment results. It includes spectra for the 57 substances specified in Japan's Specifications and Standards for Food Additives.



Contaminant Analysis Program

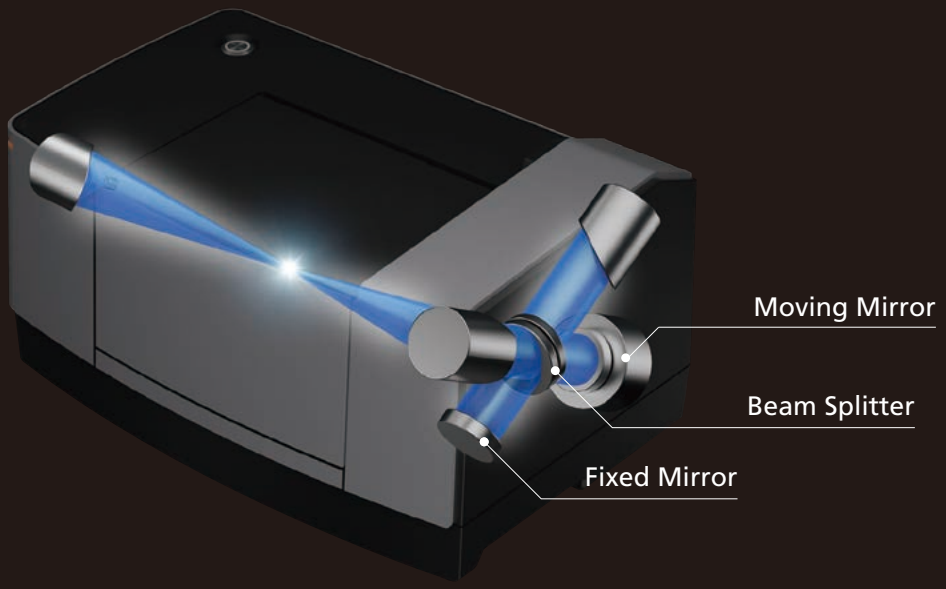
The contaminant analysis program identifies measured contaminants using Shimadzu's proprietary identification algorithm (Japanese Patent No. 5205918) in combination with a spectral library containing more than 550 spectra for substances commonly detected as contaminants. After data analysis, it automatically makes a pass/fail judgment and creates a report. Even if the contaminant is a mixture, it searches for major and minor components and displays their ranks. Since the number of components in the mixture does not need to be specified, even operators with minimal infrared analysis experience can easily analyze samples.



Shown here is an analysis of a contaminant attached to a tablet surface using the Contaminant Analysis Program. The results showed Polytetrafluoroethylene (PTFE) was the major component, and sugar and calcium carbonate were the accessory components. Since the two accessory components are often used as tablet components, it is estimated that the components were scraped up at the same time when scraping the contaminant.

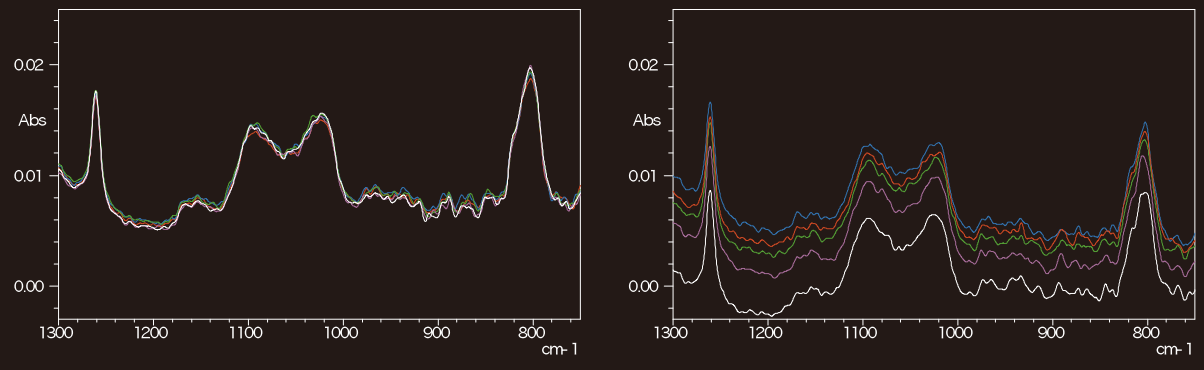
High Reliability Ensures the System Can Be Introduced with Confidence

Technology Inherited from Higher-End Models



- High stability and throughput
  (Features dynamic alignment at 5000 times per second and a high-brightness ceramic light source)
- High sensitivity comparable to general-purpose models (IRSpirit-T)
  (DLATGS detector with temperature control function)

The silicone oil content in the paraffin oil (1.0%) was obtained by repeating the single-reflection ATR method for five times. Data obtained using the DLATGS detector with temperature control is shown on the left and data obtained using the DLATGS detector without temperature control is shown on the right. The internal heat in the instrument and the environment temperature caused large baseline data fluctuations without temperature control. In contrast, using the detector with temperature control resulted in highly repeatable data.



ATR Spectrum of Silicone Oil Content in Paraffin Oil (measurement repeated five times)
Left: DLATGS detector with temperature control
Right: DLATGS detector without temperature control

High Reliability Due to Humidity-Resistant Design

The robust optics are designed to ensure the system can be used reliably even under harsh temperature and humidity conditions.

- Optics sealed in an aluminum die cast body
- Status monitor function features electrical and paper-based indicators.
- Beam splitter includes a humidity-resistant coating.
- Select from a KBr window (to 70% RH) or a KBS-5 window (to 90% RH) which both include a humidity-resistant coating.*

*No condensation

KBr Window (Selectable)

KRS-5 Window (Selectable)

Aluminum Die Cast Interferometer

Instrument Status Monitoring

Instrument status is automatically verified during start-up and results are saved in a report. This feature is especially convenient for instrument management. Pharmacopoeia-compliant programs* convenient for routine inspections are also included.

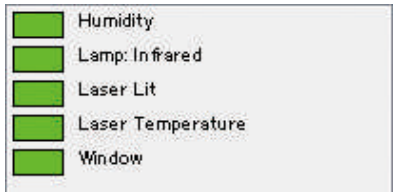
* See description of identification test program on page 7.

■ Self-diagnostic function

An automatic self-diagnostic function inspects the signal system and optics during instrument initialization. It obtains a variety of information and automatically outputs the results in one file, making instrument management easy.
The instrument status history can also be confirmed.

■ Status monitor function

This function continuously monitors and manages information about the light source, semiconductor laser, humidity inside the instrument, the window plate connection (interlock), and accessories.

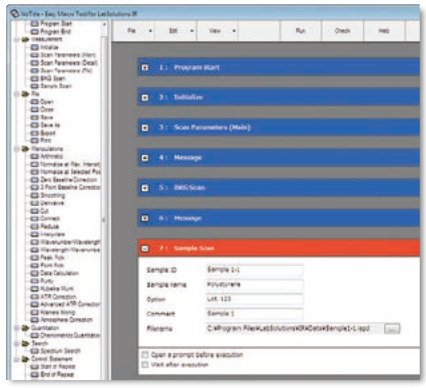


Macro Functions Provide Automation and Labor-Savings

LabSolutions IR automates routine work, such as scanning, data manipulation, reporting, identification tests, and contaminants analysis. Launch programs from the Launcher or your PC.

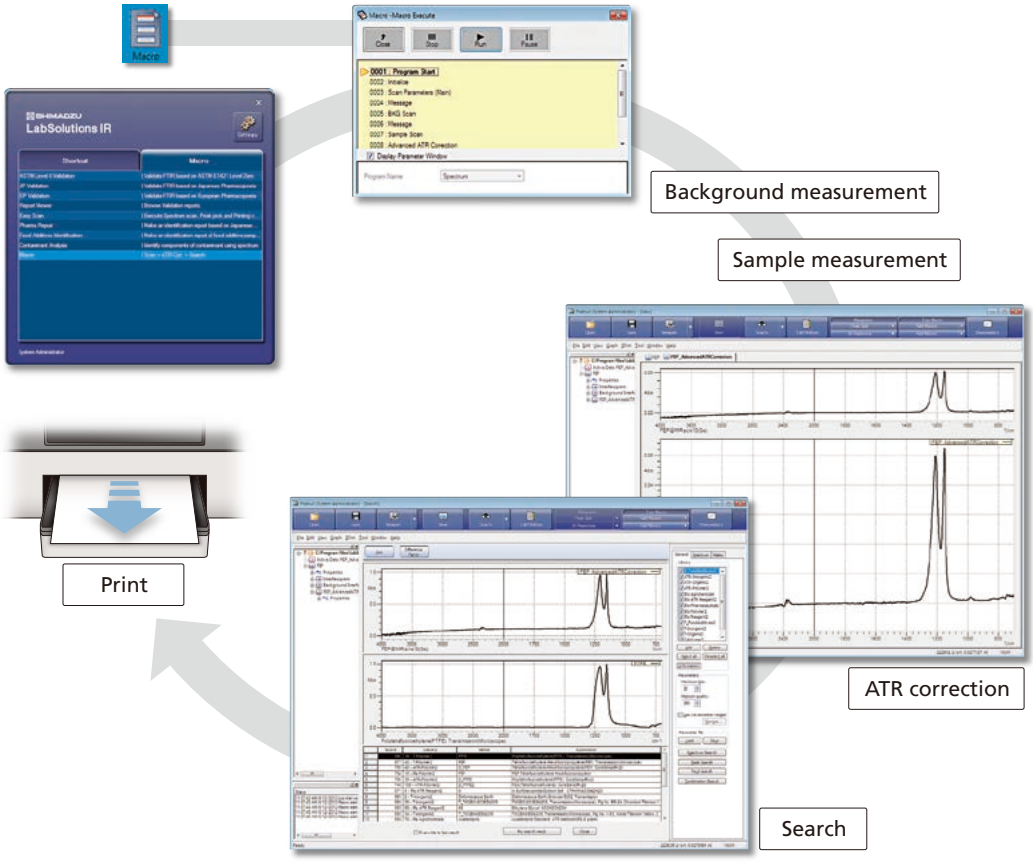
Easy Macro – Just a Single Click Launches Routine Work

The "Easy Macro" function will create macros that are suitable for routine work, particularly when repetitive operations are used. The macro builder allows macros to be constructed by simply selecting and aligning operations from a list. Once constructed, the macros can be registered with the Launcher and Desktop for quick execution. Operators who are not familiar with FTIR can easily operate the instrument.



Easy Macro Operations

- Initialization of FTIR, configuration of scan parameters, spectrum measurement
- Data manipulations, search, quantitation, printing
- Repeat measurements, displaying messages, alarm sounds, external program execution



Hardware Options

QATR-S

The QATR-S is a single-reflection ATR measurement attachment. The prism is made of diamond, so the measurement wavenumber range is up to 400 cm^{-1} (wide-band). With a liquid sample, simply place a drop on the prism to measure spectra. For other samples, clamp the sample closely against the prism surface before measurement on the sample surface. Large samples (with a large area) can be measured without cutting. The incident angle is 45 degrees. Three prism materials are available: diamond (high-throughput), germanium (Ge) or zinc selenide (ZnSe). The Ge prism is best suited for samples with a high refractive index.
Functionality is included for automatically recognizing attached accessories.

DRS-8000A

(P/N 206-62301-58)

Although powder samples are mixed with KBr, as with the KBr pellet method, the DRS-8000A analyzes the samples in their original state; creating pellets is not necessary. For plastic moldings, emery paper attached to the SiC sampler (P/N 200-66750-01) scrapes off part of the surface, forming a powdered sample that can be analyzed. Easily obtain diffuse reflectance spectra similar to transmittance spectra using the built-in Kubelka-Munk conversion in LabSolutions IR software.
Functionality is included for automatically recognizing attached accessories.

SiC sampler

MHP-1

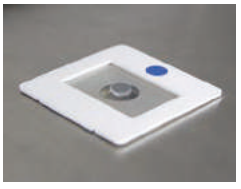
(P/N 200-66747-91)

This is a compact, inexpensive hand-driven press used to produce 4 mm dia KBr pellets. A pellet produced in the frame is directly measured using the dedicated holder, which ensures exceptional simplicity of operation. No dies or vacuum pump are required.

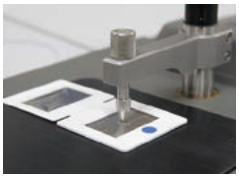
EDXIR-Holder

(P/N 221-25890-41)

This foldable holder consists of an adhesive layer with samples attached and polypropylene film designed for X-ray fluorescence analysis. It provides measurement by keeping the samples in the holder with EDX and FTIR. When using EDX for measurement, close the holder and place the polypropylene film directly to the irradiation side (downside). When using FTIR for measurement, open the holder and press the samples attached to the adhesive layer against the ATR prism.
Close the holder after the measurement and it can be used as a sample stocker.



Measurement with EDX



Measurement with FTIR

Software Options

Contaminant Library for LabSolutions IR

(P/N 206-33179-91)

This is Shimadzu's latest original library. It is an effective tool for analyzing contaminants in tap water and food. In addition to containing information on actually sampled contaminants and information about water supply maintenance parts commercially available in Japan, the library also includes X-ray fluorescence profiles (PDF files) to significantly improve the accuracy of contaminant searches. Unlike existing libraries, this contains data on mixed compounds and incorporates all the depth of knowledge and experience needed to make qualitative assessments.

Thermal-Damaged Plastics Library*

(P/N 206-33039-91)

Unlike existing libraries, this library contains data of degraded plastics that have been oxidized by heating. The library demonstrates its effectiveness when the contaminants include degraded substances, as is often the case.

* The library was compiled by Shimadzu Corporation from spectra measured and acquired by the Hamamatsu Technical Support Center, Industrial Research Institute of Shizuoka Prefecture.

Time Course Software

(P/N 206-74558-91)

The time course program is used to collect spectra in regular intervals and creates a time course dataset used to follow reactions as a function of time. Changes in peak height and peak area can be used to calculate values related to reaction kinetics. Time course information is saved and displayed in 3D (bird's eye view) or in a contour plot. It can be recalculated by modifying parameters.
The scan interval is dependent on resolution and number of scans. Maximum measurement time is 48 hours but it depends on scan parameters. The time course software includes a 3D Processing program.

EDXIR-Analysis Software

(P/N 206-33175-91)

EDXIR-Analysis software is specially designed to perform qualitative analysis using data acquired by an energy dispersive X-ray (EDX) fluorescence spectrometer and a Fourier transform infrared spectrophotometer (FTIR). This software is used to perform an integrated analysis of data from FTIR, which is excellent at the identification and qualification of organic compounds, and from EDX, which is excellent at the elementary analysis of metals, inorganic compounds and other content. It then pursues identification results and the degree of matching.

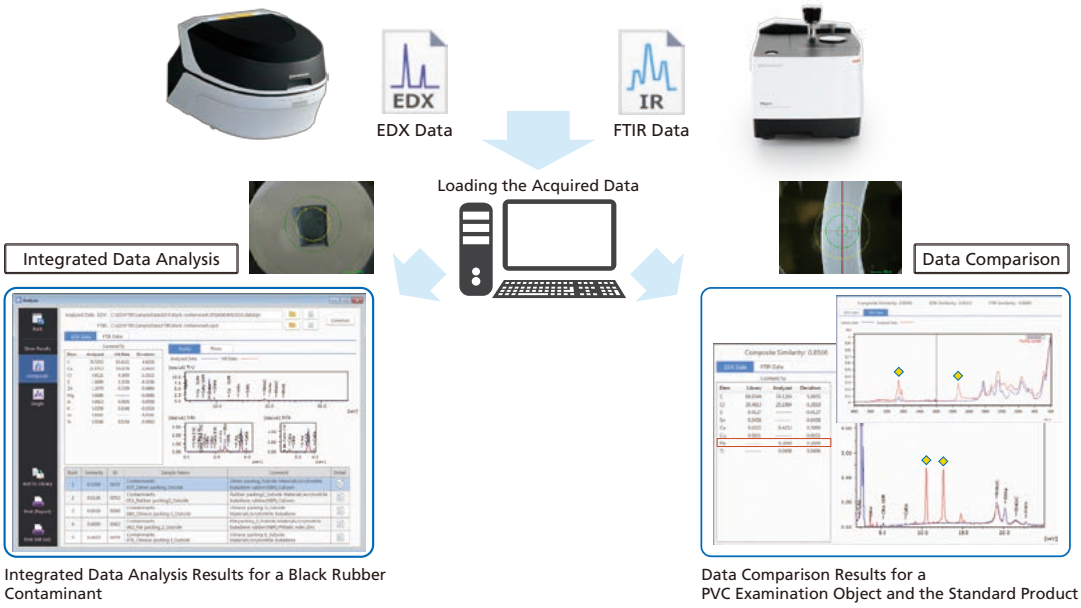
To perform qualitative analysis automatically, simply click "Analyze Both Data" and select the EDX/FTIR data*1. This heightens the efficiency of data analysis and provides strong support for contaminant analysis. In addition to a list of hits, the integrated data analysis results show EDX profiles and FTIR spectra found as hits from the library. If the user wishes to browse the respective data analysis results, they can be checked by clicking "Single". In addition, with the data comparison function, which calculates the degree of matching between the actual measured data and the data registered in the library, the software can be used for countermeasures against "silent change"*2 and for other confirmation tests.
Clicking the "Print" button prints the results in a fixed format and also saves them in Word format*3.

It can also be used to perform EDX or FTIR data analysis on its own. The library used for data analysis (containing 485 data files) is original to Shimadzu, and was created through cooperation with water supply agencies and food manufacturers. Additional data can be registered to the library, as can image files and document files in PDF format. It is also effective for the linked storage of various types of data as electronic files.

*1: Using the EDX profile, data are classified as inorganic, organic, and mixture. Integrated data analysis is performed by applying priority levels to each classification. (Patent pending)
*2: A term used in Japan to indicate changes to materials by suppliers without the knowledge of the manufacturers.
*3: Microsoft Word must first be installed.

Integrated Analysis of Contaminant Data and Data Comparisons for Confirmation Tests

The examples here show an integrated analysis of black rubber contaminant data and a data comparison for a polyvinyl chloride (PVC) examination object and the standard product. From the integrated data analysis results, it is evident that the black rubber contaminant is acrylonitrile-butadiene rubber (NBR), which contains calcium carbonate and zinc stearate. In addition, from the data comparison, the degree of matching between the PVC examination object and the standard product is 0.8506. Lead (Pb) and acrylic were detected from the EDX and FTIR data, but not detected in the standard product. Accordingly, it is surmised that the examination object contains components different to those in the standard product.

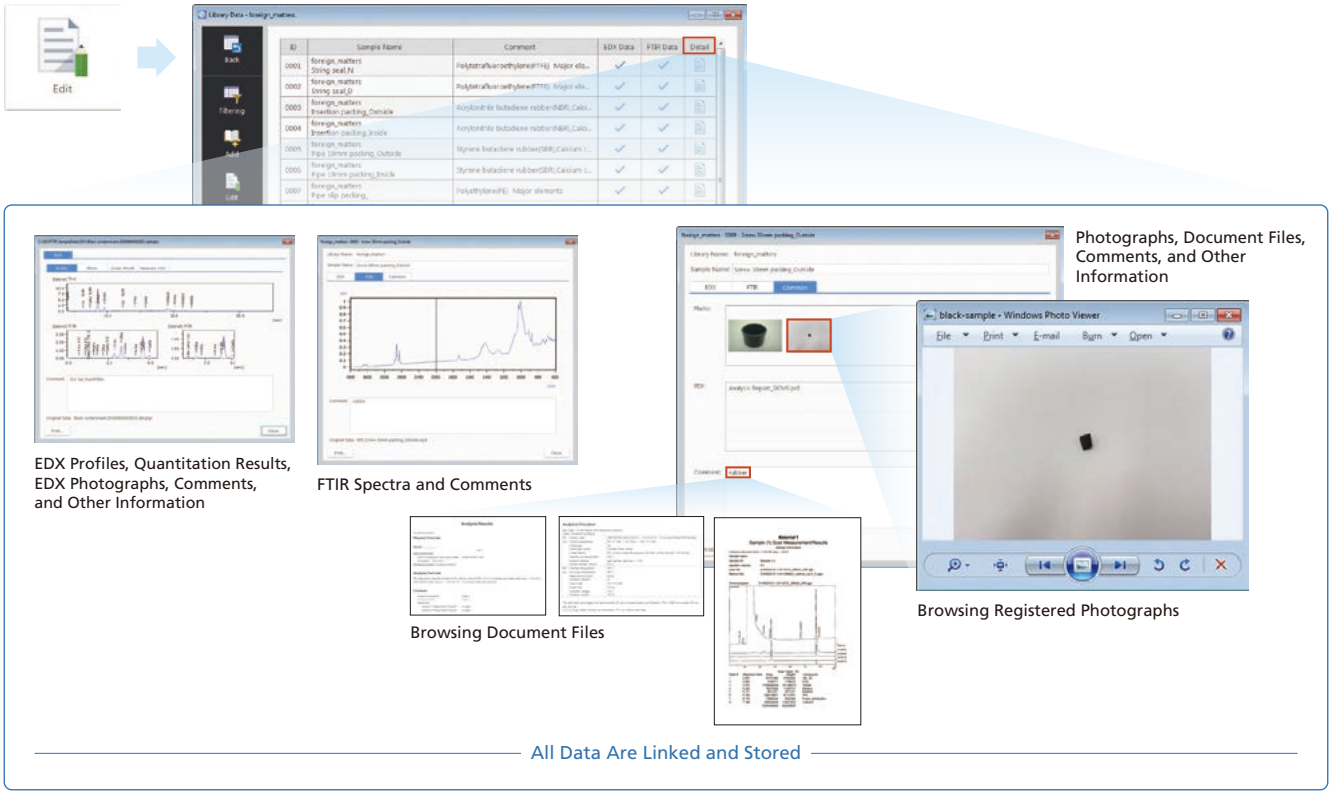


Integrated Data Analysis Results for a Black Rubber Contaminant

Data Comparison Results for a PVC Examination Object and the Standard Product

Data Browsing and the Registration, Editing, Deletion of Data, Images, Document Files

By clicking "Edit" and selecting an existing library, the data, images and documents registered in the selected library can be browsed. Data can be newly registered, edited and deleted. A new library can also be created.
In addition, if data for a sample were acquired by instruments other than EDX and FTIR instruments (such as a chromatograph, mass spectrometer, or surface observation system), it can be converted into PDF format and then registered, enabling linked storage to the EDX/FTIR data.



Solutions Achieved with LabSolutions IR

Reliable LabSolutions Software

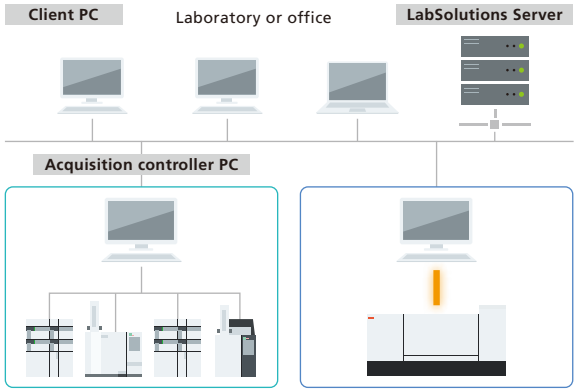
In addition to LabSolutions IR, which provides basic functionality, Shimadzu also offers LabSolutions DB IR and LabSolutions CS IR to meet the requirements of ER/ES regulations.

LabSolutions DB IR

LabSolutions DB IR allows for secure data management by integrating a data management function with LabSolutions IR. Compliant with ER/ES regulations, the software is optimally configured for customers using a PC. It is recommended for facilities that do not require network connections and want to be ER/ES compliant.

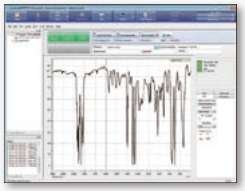
LabSolutions CS IR

LabSolutions CS, which is freely accessible to the analysis network, can be connected to IR, eliminating the need for connecting a PC to the instrument. Since all the data are managed on a server, LabSolutions CS IR can be read from any personal computer on a network. With terminal service, LabSolutions IR can be controlled from a client PC without installing LabSolutions IR on it. It is recommended for facilities that have a large number of users, manage data in a database, and want to be ER/ES compliant.



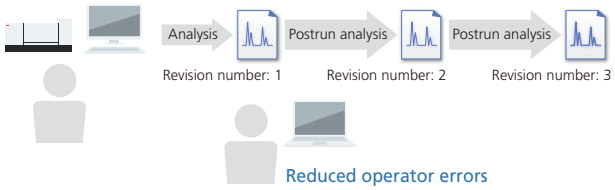
Name	LabSolutions IR	LabSolutions DB IR	LabSolutions CS IR
Data management method	Measured data files are saved and managed in folders on the PC.	Measured data files are saved and managed in the LabSolutions database.	
Data references	The software references files on drives or in folders on the PC.	The software references files in the database.	
LabSolutions database	Unavailable	Available (The database resides on a local PC)	Available (The database resides on a server)
User administration	Available		
Rights group administration	Available		
Project administration	Unavailable	Available	
Standalone/network	Either can be used.	Only the standalone configuration can be used.	Only databases on the network can be used. (LabSolutions IR data can be viewed using the database manager on a PC set up for viewing purposes. Note that LabSolutions IR must be installed on the PC used for viewing.)
Data backup	Performed on a file-by-file basis using Windows Explorer.	Performed for each database.	

Operate with LabSolutions, Shimadzu's reliable and popular workstation used in chromatography and spectroscopic analysis.


Spectral Measurement Screen

Database Management Prevents Mistakes

With LabSolutions DB IR and CS IR, the analysis data are managed securely by the database. Overwriting, deletion and other mistakes typical of data file management do not occur. In addition, when postrun analysis is performed using the acquired data, postrun analysis data revision numbers are automatically assigned, preventing the accidental overwriting of raw data.

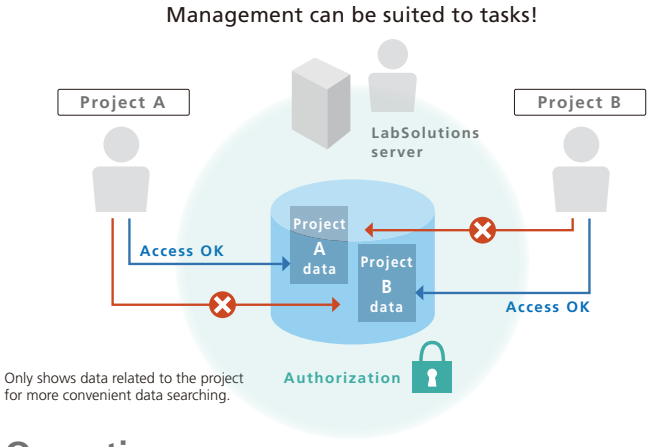


Solid Security

An audit trail to ensure the reliability of data and document e-mail transmission functions when any event occurs in the system can be set up. User accounts are managed using passwords, where password length, complexity and term of validity must satisfy specified requirements. It is also possible to set lockout functions to prevent illegal access, and set a registered user's deletion and change. In addition, a box can be selected to prevent overwriting a data file, and outputting an item to a report can also be performed.

Pertinent Information is Managed for Every Project

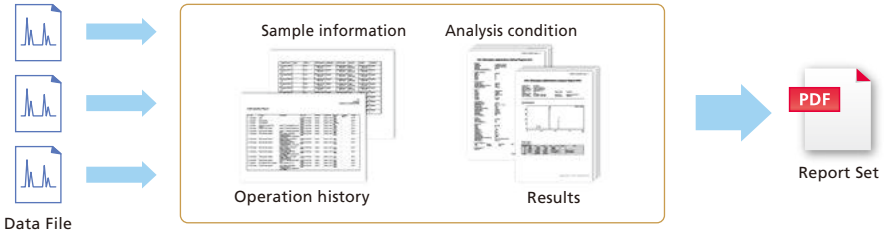
LabSolutions DB IR and CS IR provide a project management function enabling management suited to tasks and system operations. This function enables equipment and user management, security policy, and data processing to be set on a project by project basis, thereby improving the efficiency of data searches and management tasks.



Visualization of the Sequence of Analysis Operations

Creating a report set* provides visibility of the individual analytical operations involved in the overall analytical process. When analytical operations are visible, it is easier to check for operating errors, which helps improve the efficiency and reliability of checking processes.

* Report sets include test methods and test results for a series of samples analyzed, and also a corresponding operation log (a record of all operating events from login to logout), which is automatically extracted from the data and summarized in a single report.



Specifications

Hardware

Interferometer	Michelson interferometer with 30° degree angle of incidence Dynamic alignment Sealed dry interferometer
Optical system	Single-beam optics with built-in temperature and humidity sensors
Detector	IRSpirit-T model: DLATGS detector with temperature control IRSpirit-L model: LiTaO3 detector
Beam splitter	Germanium-coated KBr
Light source	High-energy ceramic (3 years guaranteed)
Max Resolution	0.9 cm^{-1}
Sample compartment	Equipped with automatic accessory recognition mechanism 200(W) × 140(D) × 100(H) mm Center focus
Dimensions	390(W) × 250(D) × 210(H) mm
Weight	8.5 kg

Software

Operating system	Windows 7 Professional 32/64-bit edition Windows 10 Pro 64-bit edition
Interface	USB 2.0, USB 3.0
Validation program	Complies with Chinese, European, US, and Japanese Pharmacopoeias and ASTM
GLP/GMP support	Tree-structured audit trail function Recording of operation logs and data logs (history) Saving by overwriting the same filename is prohibited
Security functions	Coordination with LabSolutions security functions User-group-based privilege settings

Other

Required environment	Operating guaranteed temperature: 10 to 35°C Humidity: 20-70% (KBr window model) 20-90% (KRS-5 window model) with no condensation
Power requirements	AC 100 to 240V, 50/60 Hz, 75 VA (when used) or 7 VA (standby)

Transporting and Moving the System:
Do not apply shocks such as vibrations or drops to the system during transportation. When you move the IRSpirit over a long distance, such as moving to another building, it must be packed into the packing box when IRSpirit arrived at your facility. The performance deterioration caused by the transportation is not covered by warranty.

This product conforms to Shimadzu's Eco-labeled designation.
Energy savings: 34% reduction as compared to the previous model*
Space savings: 76% reduction of weight as compared to the previous model*
70% reduction of installation area as compared to the previous model*
*: IRAffinity-1S

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Fourier Transform Infrared Spectrophotometer

IRSpirit

IRSpirit, Ready to Run

IRSpirit offers the highest S/N ratio in its class and high reliability using the technology inherited from the high-end model. The dedicated IR Pilot program simplifies routine analyses for all users.

Space-Efficient with High Expandability

Despite its compact size, the sample compartment width is the same as on higher-end models, allowing it to easily accommodate both Shimadzu and third-party accessories. That means this single system can be used for a wide variety of applications.

Samples can be measured with the unit positioned horizontally or vertically for sites with only a narrow space available.

The start switch is accessible and the humidity indicator is visible from both directions.

Large Sample Compartment in a Small Body

With the widest sample compartment in its class, it easily accommodates Shimadzu and third-party accessories.

Dedicated IR Pilot Program

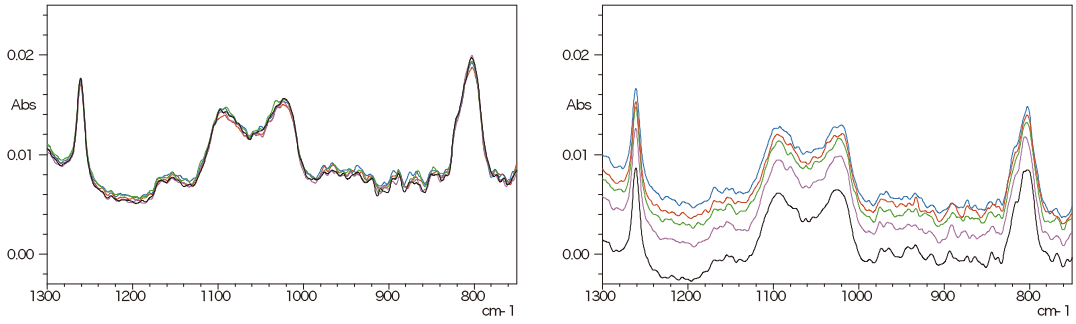
The newly developed dedicated program (IR Pilot) features 23 standard application workflows within four programs: identification test, contaminant analysis, quantitative analysis and film thickness calculation, which can be conducted without setting parameters. By simply selecting the analysis purpose and accessory, analysis can be performed, which makes it easy for operators with minimal FTIR experience to obtain results.

* Quantitative analysis function cannot be used when using IR Pilot with LabSolutions DB/CS.

High Reliability Ensures the System Can Be Introduced with Confidence

IRSpirit offers the highest S/N ratio (30,000:1*) in its class using the technology inherited from the high-end model. Furthermore, the robust optics are designed to ensure the system can be used reliably even under harsh temperature and humidity conditions.

*: DLATGS detector with KBr window



DLATGS Detector with Temperature Control

DLATGS Detector without Temperature Control

ATR Spectrum of Silicone Oil Content in Paraffin Oil (measurement repeated five times)

The internal heat in the instrument and the environment temperature caused large baseline data fluctuations when using a detector without temperature control. In contrast, a detector with temperature control provided repeatable data.

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Application News

No. A549

Spectrophotometric Analysis

Contaminant Analysis on Tablet Surface Using the IRSpirit

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, designed for maximum flexibility, 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 interface within LabSolutionsIR, has 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 demonstrates the efficiency of the IRSpirit and QATR-S accessory in analyzing a contaminant on the surface of a tablet.

R. Fuji

Fig. 1 Picture of the IRSpirit Compact FTIR

Contaminant Analysis in Pharmaceutical Manufacturing

Despite the rigorous controls in pharmaceutical manufacturing, surface defects are sometimes detected during visual examination of tablets. To resolve contamination issues, it is important to know if the contaminant is intrinsic, such as an undesirable aggregation of ingredients, or alternatively, something that has its origins entirely outside of the manufacturing process. There are several experimental approaches to analyze a surface contaminant, such as removing the area for further analysis. The quality of results often depends on the skill and judgement of the analyst in extracting the contaminant area, as well as the potential for introducing further contamination. Another approach is to investigate the contaminant in situ using FT-ATR. The effectiveness of this approach can be limited for sub-surface contaminants as ATR measures only the material in direct contact with the crystal. The size and shape of the sample will also impact the results, ideally the contaminant would completely cover the 1 mm diameter sample area of the ATR crystal.

Surface Contamination on Tablet - Comparison of Sampling Methods -

This article compares the results of contaminant spectra taken with the sample in situ versus removed by scraping. Fig. 2 shows a contaminant (simulated for the purposes of this study) on the surface of a tablet. The area highlighted by the red circle is approximately 1.2 mm in size, and is exposed on the surface of the tablet. These characteristics (size and surface location) make this a good candidate for ATR measurements. The sample is somewhat irregular in shape though, and may not entirely covert the ATR crystal during a measurement. As ATR is non-destructive though, it is possible to compare the two sampling methods. After acquiring ATR spectra, the sample can be scraped, and re-analyzed.

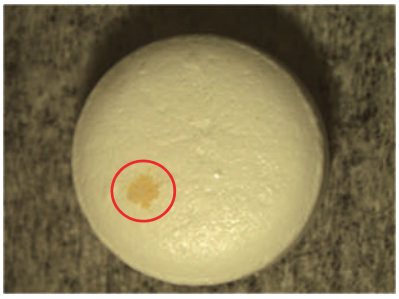

Fig. 2 Picture of Contaminant on Tablet Surface

QATR-S Dedicated ATR Accessory

The QATR-S single bounce ATR accessory has been designed specifically for the IRSpirit (Fig. 3), with easily user-swappable diamond and germanium crystals. FT-ATR spectroscopy is widely used for the analysis of solid and liquid samples because it greatly simplifies sample preparation. By making firm contact between the sample and the crystal, IR spectra of bulk materials can be acquired without the need for "diluting" the sample with KBr, for example.

Fig. 3 QATR-S Accessory Installed in IRSpirit Sample Compartment

Table 1 shows the experimental parameters used to collect data for contaminant analysis. The wideband diamond crystal was used.

Table 1 Measurement Conditions

Instrument	:	IRSpirit-L (KBr window), QATR-S (Wideband diamond disk)
Resolution	:	4 cm^{-1}
Accumulation	:	45
Apodization	:	Sqr Triangle
Detector	:	$LiTaO_3$

In Situ Contaminant Analysis via FT-ATR

The spectrum acquired from the contaminant is shown in as the black trace in Fig. 4. The library search results highlight two likely sample components, acetylsalicylic acid (green spectrum) and acetaminophen (red spectrum).

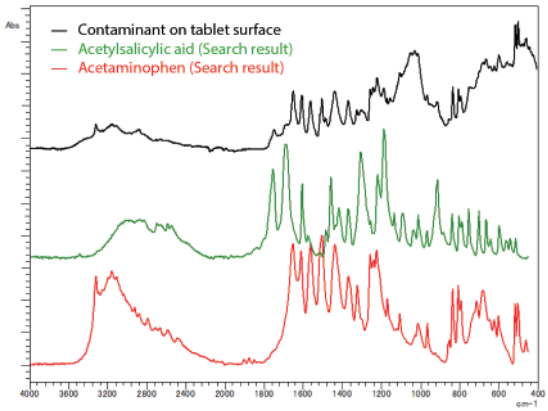

Fig. 4 Analysis of In Situ Contaminant Using QATR-S

FT-ATR Analysis of Removed Contaminant

Following the in situ analysis, the contaminant was scraped from the tablet, and the resulting powder was again measured with FT-ATR. A portion of the sample matrix was similarly removed and measured. Fig. 5 and Fig. 6 show infrared spectra of the contaminant and tablet, respectively, along with the search results for each. This analysis shows that the contaminant is acetaminophen, whereas the tablet matrix is acetylsalicylic acid.

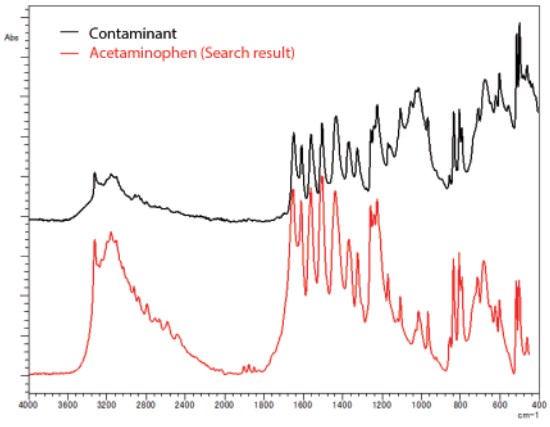

Fig. 5 Contaminant Spectrum and Search Result – Destructive Sampling

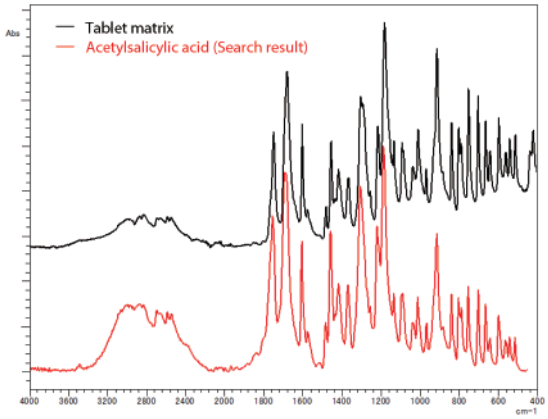

Fig. 6 Tablet Matrix Spectrum and Search Result – Destructive Sampling

Conclusion

A tablet surface contaminant was analyzed using the IRSpirit with the QATR-S accessory. As FT-ATR is a non-destructive measurement technique, it was possible to compare results of in situ sampling to a subsequent destructive contaminant removal approach. The in situ approach showed a mixture of components, mostly acetaminophen with a slight contribution from acetylsalicylic acid. The results of the destructive sampling approach clearly separated the contaminant, acetaminophen, from the sample matrix acetylsalicylic acid. In this example, because of the relatively small size and irregular shape of the tablet contaminant, the destructive sample approach gave a more robust answer to the chemical identification question. For both sampling methods, it was easy to determine that the contaminant was something intrinsic to the manufacturing process, and not an environmental or external contaminant.

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Application News

No. A546

Spectrophotometric Analysis

Identification of Contaminant Found in Resin Production Process Using IR Pilot

The Shimadzu IRSpirit launched recently is a compact Fourier transform infrared spectrophotometer characterized by the highest signal to noise ratio and the highest maximum resolution in its class (Fig. 1). The dimensions of the body are W390 × D250 × H210 mm and less than A3 in size. The IRSpirit adopts a unique design which allows not only lateral installation but also longitudinal installation so that it can be installed in a narrow space in width. The IRSpirit also has another feature that it can mount the existing accessories such as a single reflection type attenuated total reflection attachment and a diffuse reflection attachment and the commercially available accessories in addition to the accessories for the transmission measurement such as a demountable cell and a KBr pellet holder.

A dedicated program named IR Pilot to assist the analysis, which allows four measurement items - identification test, contaminant analysis, quantitative analysis and film thickness determination - to be easily operated, is equipped in the LabSolutions IR software which controls the IRSpirit and executes data manipulations. By using the IR Pilot, successive operations of measurement, analysis and print can be orderly performed following the on-screen instructions. The IR Pilot is comprised of twenty-three kinds of the dedicated program of which the often-used program up to four kinds can be registered in its main menu.

This article introduces the contaminant analysis using the IR Pilot.

H. Abo

Fig. 1 External View of Shimadzu IRSpirit compact FTIR

Procedures of Contaminant Analysis

The contaminant analysis using FITR is conducted in a variety of industries and essential to identify the pathway and the source of the contamination.

In general, the contaminant analysis is performed according to the procedures written below.

1. Select a measurement technique suitable to a sample form.
2. Set the FTIR measurement parameters.
3. Measure a background spectrum.
4. Set a contaminant to an instrument.
5. Measure it.
6. Execute data manipulations if needed.
7. Execute spectrum search.
8. Print the results.

There are some settings and selections to note for such successive operations. The IR Pilot supports them. The IR Plot allows the selection of a method and the setting of parameters to be completed only following the on-screen instructions and enables the next operation to be understood.

Table 1 shows the conditions used for the measurement.

Table 1 Measurement Conditions

Instrument	: IRSpirit-L (KBr window), QuestATR (Wideband diamond disk)
Resolution	: 4 cm^{-1}
Accumulation	: 45
Apodization	: Sqr Triangle
Detector	: $LiTaO_3$

Contaminant Analysis Using the IR Pilot

The contaminant analysis was performed using the IR Plot. A white contaminant found in a resin production process was measured. After starting the program, "Spectrum" program in the LabSolutions IR is displayed on the screen and the IRSpirit is automatically initialized.

"Main Menu" is displayed as the initial screen of the IR Pilot. Then "Contaminant Analysis" in it is selected (Fig. 2).

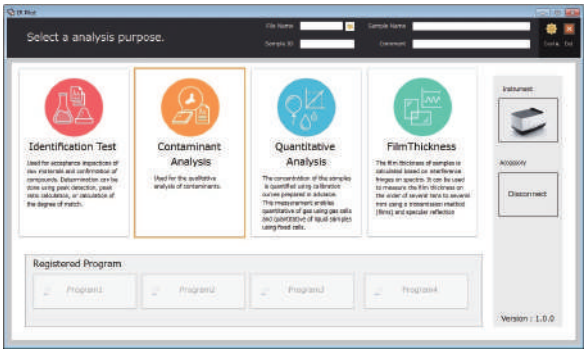

Fig. 2 Main Menu

After executing "Contaminant Analysis", a screen to select a measurement technique is displayed (Fig. 3). When the ATR measurement is performed, "Yes" is chosen. If another technique is used, "No" is selected. If the ATR measurement instrument is automatically recognized, ATR spectroscopy will be selected automatically, and Fig. 3 screen will not be displayed.

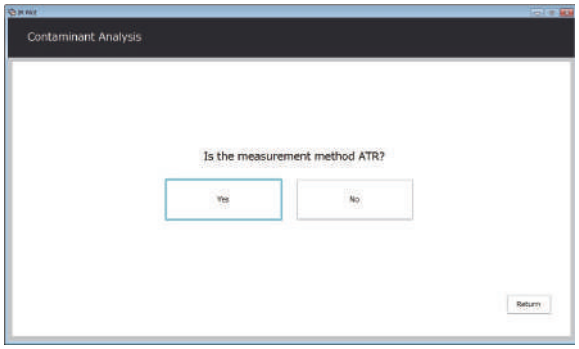

Fig. 3 Screen to Select Measurement Technique

Next, a prism used for the measurement is selected. A diamond prism is often used for ATR spectroscopy. However, when a substance with high refractive index such as black rubber is measured with a diamond prism, its peaks are often distorted by anomalous dispersion of refractive index. In such a case, a germanium prism is used to avoid the distortion. The operable wavenumber range depends on a using prim. The IR Plot automatically set the wavenumber range suitable to a selected prism (Fig. 4). In this article, a diamond prism was used for the measurement.

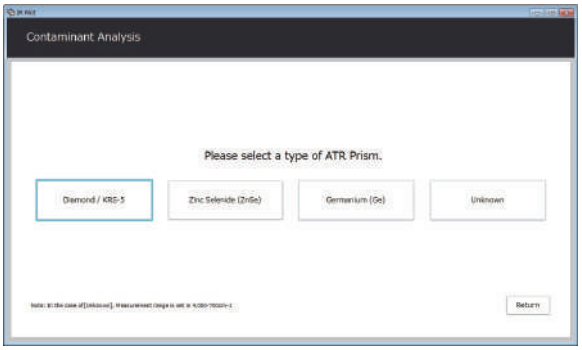

Fig. 4 Screen to Select Prism

After selecting a prism, a screen to select the execution of the background measurement is displayed. After confirming that a sample is not on a prism, the background measurement is performed.

When the background measurement is completed, a screen to indicate the sample setting is displayed. After tightly holding a sample on a prism, the sample measurement is performed by pressing "OK" button.

After measuring the sample, a screen to select the execution of ATR correction is displayed. If needed, "Yes" is chosen to execute it. ATR correction requires the input of some parameters such as the incident angle and the number of reflection which are written in an instruction manual of an ATR attachment (Fig. 5). The input of the refractive index of a sample is also necessary. In general, since the refractive index of an organic compound is in the range from about 1.4 to 1.7, 1.5 is input if the refractive index of the sample is unknown. Although the refractive index of the prism is also needed for ATR correction, it is automatically set when the prism is selected. "Execute" button is pressed to execute ATR correction.

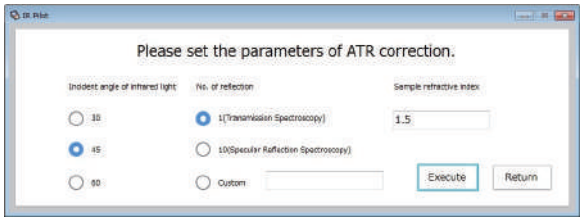

Fig. 5 Screen to Input Parameters for ATR Correction

Then a screen to select the execution of spectrum search for a measured spectrum is displayed. If needed, "Yes" is chosen to execute it. The setting of the search parameters is not necessary because it is automatically done. After the spectrum search, the result can be printed. If the print of the result is needed, "Yes" is chosen in the screen to execute the print. Fig. 6 shows an example of the print. An infrared spectrum of a sample is printed with infrared spectra of the first to third match in the search result to compare it with them. The list up to 20th match is also printed. The search result indicated that the measured contaminant was a kind of protein originating from a human being.

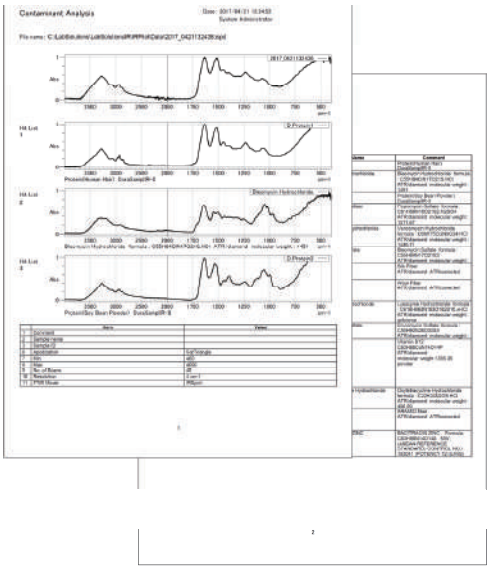

Fig. 6 Example of Print

Finally, a screen to register the program shown in Fig. 7 is displayed. The program following a series of operations for the contaminant analysis written in this article can be registered in the main menu. The registration allows the next measurement to be more easily performed because the number of displayed screen is reduced.

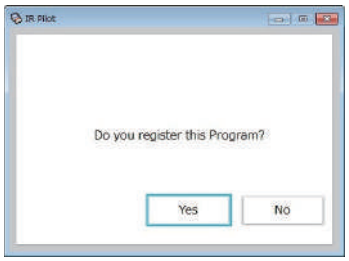

Fig. 7 Screen to Register Program

Conclusion

The contaminant analysis was performed using the IR Pilot. The IR Pilot can execute successive operations of the setting of measurement parameters, the background measurement, the sample measurement, the data manipulation, the spectrum search and the print automatically and facilitates the contaminant analysis. The IR Pilot allows a beginner of FTIR and a user conducting the routine measurement to use the Shimadzu IRSpirit more comfortably according to the proper procedures. Please try to use it.

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Application News

No. A548

Spectrophotometric Analysis

Thickness Determination of Film and Coating on Inside of Can Using IR Pilot

The Shimadzu IRSpirit launched recently is a compact Fourier transform infrared spectrophotometer characterized by the highest signal to noise ratio and the highest maximum resolution in its class (Fig. 1). The dimensions of the body are W390 × D250 × H210 mm and less than A3 in size. The IRSpirit adopts a unique design which allows not only lateral installation but also longitudinal installation so that it can be installed in a narrow space in width. The IRSpirit also has another feature that it can mount the existing accessories such as a single reflection type attenuated total reflection attachment and a diffuse reflection attachment and the commercially available accessories in addition to the accessories for the transmission measurement such as a demountable cell and a KBr pellet holder.

A dedicated program named IR Pilot to assist the analysis, which allows four measurement items - identification test, contaminant analysis, quantitative analysis and film thickness determination - to be easily operated, is equipped in the LabSolutions IR software which controls the IRSpirit and executes data manipulations. By using the IR Pilot, successive operations of measurement, analysis and print can be orderly performed following the on-screen instructions. The IR Pilot is comprised of twenty-three kinds of the dedicated program of which the often-used program up to four kinds can be registered in its main menu.

This article introduces the film thickness determination using the IR Pilot.

Y. Suzuki

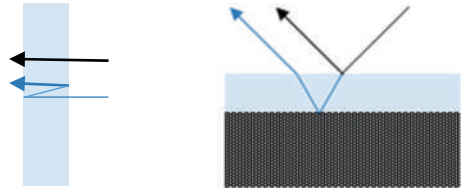
Fig. 1 Shimadzu IRSpirit compact FTIR

Principle of Film Thickness Determination

A variety of films are used in products used in daily life. For example, a multi-layer film used for the food packing laminates films to allow functions such as moisture impermeability and blocking performance against gases and light. For a touch panel in a smartphone, films providing the antireflection effect and the electric characteristic are laminated.

The film thickness determination performed by using interference fringes appearing in an infrared spectrum is one of the determination methods. Since infrared light doesn't damage a sample, the measurement can be conducted without destroying and touching it.

In the case of the transmission measurement of a film, a beam transferring to the direction of travel again after being reflected inside a film interferes with an incident beam (Fig. 2 a). For the reflection measurement of a coating on the metal surface, a beam reflected on the surface of the coating interferes with that reflected on the metal surface after transmitting the coating (Fig. 2 b).



(a) Transmission Measurement (b) Reflection Measurement

Fig. 2 Interference of Light

As a result, interference fringes whose intensities change like a sine wave as shown in Fig. 3 are observed in an infrared spectrum. Although interference fringes appear regardless of the existence or nonexistence of an absorption peak, they are clearly observed in the region where no peak exists.

Fig. 3 Interference Fringes

For both transmission spectroscopy and reflection spectroscopy, film thickness (t) is calculated using the following equation.

where n = refractive index of a sample, M = number of complete sine waves in the wavenumber range from ν_1 to ν_2 (order of interference), θ = incident angle. For transmission spectroscopy, θ is zero.

$$t = \frac{M}{2\sqrt{n^2 - \sin^2\theta}\,(\nu_1 - \nu_2)}$$

Film Thickness Determination Using the IR Pilot

After starting the IR Piot, "Spectrum" program in the LabSolutions IR is displayed on the screen and then the IRSpirit is automatically initialized. After the initialization, "Film Thickness Determination" in the main menu shown in Fig. 4 is selected as the purpose of analysis.

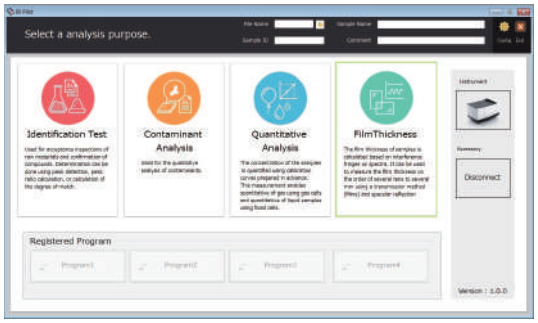


Fig. 4 Main Menu

Then incident angle and refractive index of a sample are input in a screen shown in Fig. 5 before the measurement. Zero degrees (transmission measurement) and 10 degrees (incident angle of the SRM-8000 specular refection attachment) are selectable. If a sample is measured at the other angle, the angle also can be input using "Custom".

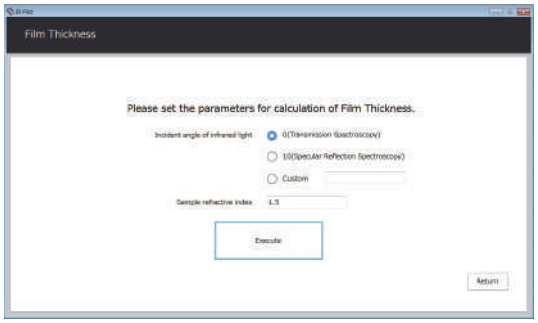


Fig. 5 Screen to Input Incident Angle and Refractive Index of Sample

At first, a polystyrene film was measured in transmission spectroscopy and its thickness was determined. The wavenumber range used for the film thickness calculation is chosen in a measured spectrum using a screen shown in Fig. 6. The wavenumber range where no absorption peaks are observed and only interference fringes appear should be selected. Here, the wavenumber range from 3833 cm^{-1} to 3205 cm^{-1} was selected.

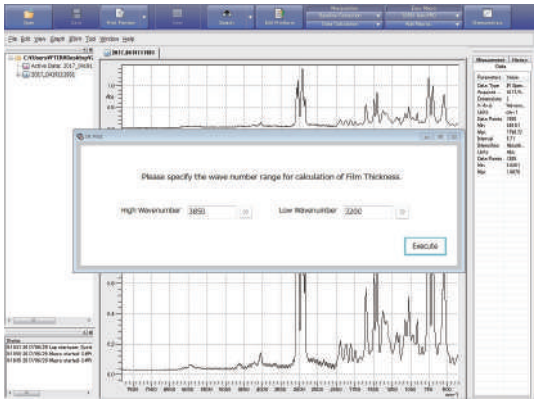


Fig. 6 Screen to Specify Wavenumber Range of Interference Fringes and Its Expanded One

The number of complete sine waves in the input range is automatically obtained. Then the film thickness is calculated using the selected incident angle, the input refractive index and the obtained number of waves. Fig. 7 shows a screen for the calculation result. The measured polystyrene film stood at 57 µm in thickness.

Fig. 7 Result of Film Thickness Determination

Next, the film thickness determination by reflection spectroscopy was performed. The coating on the inside of the bottom plate of a commercially available juice can was measured with the SRM-8000 specular reflection attachment (incident angle : 10 degrees). The infrared spectrum and the calculated result are shown in Fig. 8. The thickness was 21 µm.

A reflection measurement attachment with other incident angle is also available because the film thickness determination can be calculated with an arbitrary incident angle.

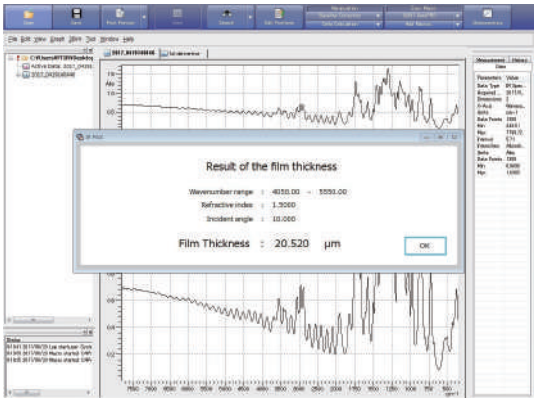


Fig. 8 Measurement of Coating on Inside of Beverage Can by Reflection Spectroscopy

Conclusion

The film thickness determination can be applied to a variety of samples such as a film, a coating and a semiconductor. An easy operation for the film thickness determination introduced in this article allows the labor-saving and efficient measurement operation. The IR Pilot can execute successive operations of the setting of measurement parameters, the background measurement, the sample measurement, the data manipulation and the print automatically and facilitate the film thickness determination. The IR Pilot allows a beginner of FTIR and a user conducting the routine measurement to use the IRSpirit more comfortably according to the proper procedures. Please try to use it.

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