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VALIDATION SOFTWARE

INSTRUCTION MANUAL

SHIMADZU FOURIER TRANSFORM

INFRARED SPECTROPHOTOMETER

Read the instruction manual thoroughly before you use the product.
Keep this instruction manual for future reference.

SHIMADZU CORPORATION
KYOTO JAPAN

ANALYTICAL & MEASURING INSTRUMENTS DIVISION

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Introduction

Read this Instruction Manual thoroughly before using the product.

Thank you for purchasing this product. This manual describes the installation, operation, usage cautions, accessories and options for this product. Read this manual thoroughly before using the product and operate the product in accordance with the instructions in this manual.

Also, keep this manual for future reference.

IMPORTANT

- If the user or usage location changes, ensure that this Instruction Manual is always kept together with the product.
- If this manual or a product warning label is lost or damaged, immediately contact your Shimadzu representative to request a replacement.
- To ensure safe operation, read all Safety Instructions before using the product.
- To ensure safe operation, contact your Shimadzu representative if product installation, adjustment, or re-installation (after the product is moved) is required.

Notice

- Information in this manual is subject to change without notice and does not represent a commitment on the part of the vendor.
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For Safety Use

IRsolution Validation software is a program to checkup and validate your FTIR. Please remember the following items when using this product.

1. Please DO NOT use this product for purposes other than what it was designed for.
2. For safety reasons, follow the procedure described in the instruction manual.
3. Obey warnings or caution descriptions.
4. DO NOT modify the product by yourself.

This instruction manual defines the levels of warning as follows;

WARNING

Indicates a potentially hazardous situation which, if not avoided, could result in serious injury or possibly death.

CAUTION

Indicates a potentially hazardous situation which, if not avoided, may result in minor to moderate injury or equipment damage.

NOTE

Emphasizes additional information that is provided to ensure the proper use of this product.

Guide for Using the Instruction Manual

This instruction manual of the Validation software for IRsolution software is comprised of this volume only.
This instruction manual describes the functions of Validation software. For general operations of the IRsolution such as measuring the sample spectra, refer to the Instruction Manual for the IRsolution software.

Contents

Chapter 1
Introduction

1.1 Introduction

This software validates the Shimadzu Fourier transform infrared spectrophotometer IRPrestige-21/IRAffinity-1/ FTIR-8000 series on the IRsolution software to control them and data processing.

This software complies with the description in 'General Test 23, Infrared Spectrophotometry' of the Fifteenth Edition of Japanese Pharmacopoeia, "Absorption Spectrophotometry, Infrared" of the European Pharmacopoeia 5.0 and ASTM (American Society for Testing and Materials) E1421-94 Level Zero. Either of polystyrene film of the standard accessory of FTIR, supplied by NIST, or installed on the Beam Switching Kit is used for the validation. The result is output to the screen and the printer. The report viewer program is included to display and print the validation result later.

Because this software works on IRsolution, the measurement and the calculation can be executed as a series of operations. It is also possible to validate using the data, which is already measured and saved from the disk.

NOTE

1. Please purchase and use the Standard Reference Material of polystyrene film supplied by NIST or its secondary standard when traceable polystyrene film is needed.
2. Only FTIR-8300/8400/8400S, IRAffinity-1 and IRPrestige-21 are adapted to the European Pharmacopoeia 5.0.
3. Please input numbers by the normal-width figure on this program.

Chapter 2
Installation of the Program

Shimadzu representative will install the Validation program. Please refer to this chapter if reinstallation is needed for some reason.

2.1 Installation of the Validation Program

The Validation program is installed when the IRsolution is installed at the same time.

2.1.1 Procedure to Build the Validation Program in the IRsolution

The Validation program is automatically installed when the IRsolution is installed, therefore the Validation program does not need to be separately installed. Information below describes the files for the Validation program.

The necessary files and the copied folder name are shown below.

File name	Type	Folder
OEMMacros.lcm	Macro container file which includes the Validation program	c:\Program Files\Shimadzu\ IRsolution
ValidASTM*.ptm	Print templates for the ASTM validation	c:\Program Files\Shimadzu\ IRsolution\Print Templates
ValidEPJP.ptm	Print template for the JP and EP validation	c:\Program Files\Shimadzu \IRsolution\PrintTemplates
ValidJP.ptm	Print template for the JP validation	c:\Program Files\Shimadzu \IRsolution\PrintTemplates
ValidEP.ptm	Print template for the EP validation	c:\Program Files\Shimadzu \IRsolution\PrintTemplates

2.1.2 Method to Edit Template File

The Validation program prints out the inspection report using the repaired print template. The print template links to files defined with the absolute drive and path. The default path, which the IRsolution is installed, is "c:\Program Files\Shimadzu\IRsolution." If the IRsolution is not installed in the default drive and/or path, please edit the template file to which the template files are installed according to the following procedure.

Procedure:

1. Start the IRsolution.
2. Click the [Print Form] tab.
3. Select the [File]-[Open]. Select "ValidASTM.ptm" and click the [OK] button. The content of "ValidASTM.ptm" is displayed on the screen.
4. Click a central part on the frame to display the [Object Property] window of the template.

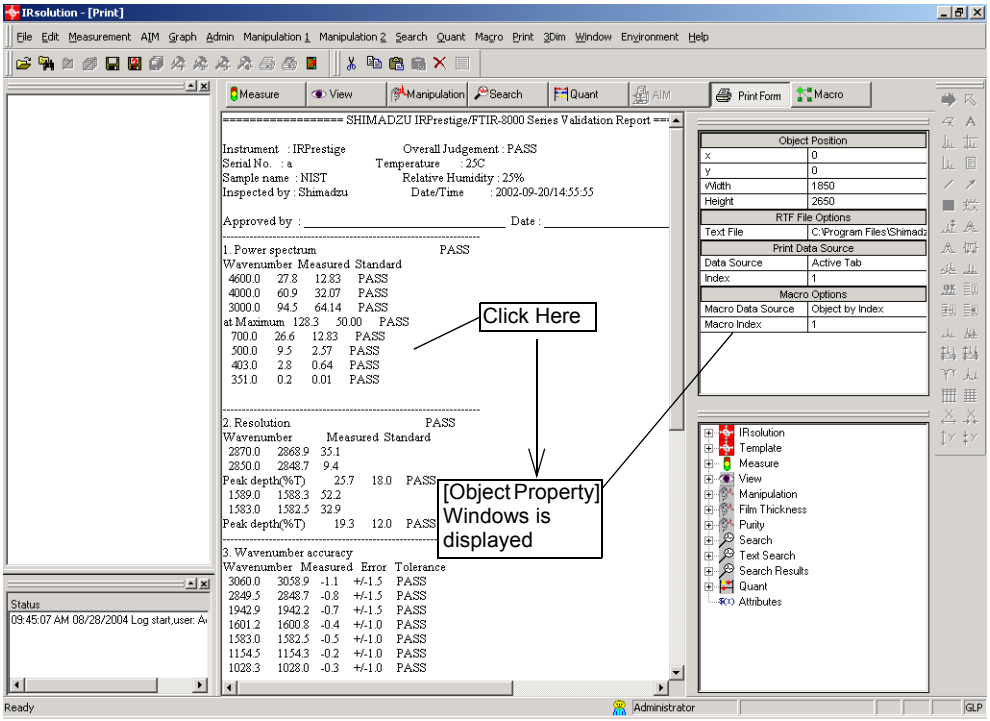


Fig. 2.1 Displayed [Object Property] Window

5. Double-click the "Text File" section at the "RTF File Options" in the [Object Property] window. The [File Open] dialog box opens and selects the "Print.txt" in the proper drive and folder for each Validation programs. Then click the [OK] button.

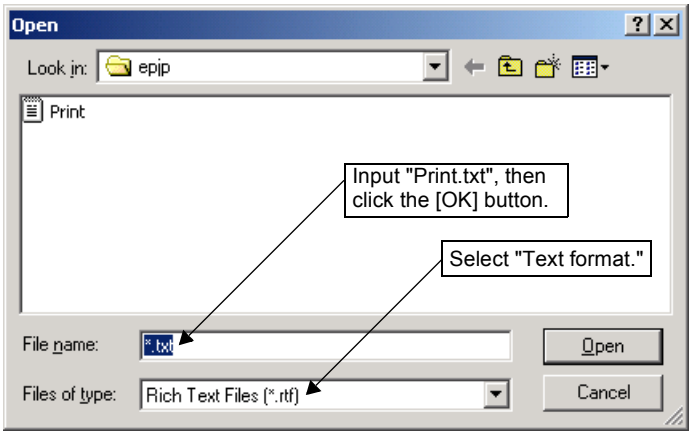


Fig. 2.2 Select the "Print.txt" File

6. The "Print.txt" file is saved as follows.

File name	Type	Folder
ValidASTM.ptm	For ASTM Validation	\(Folder which the IRsolution is installed)\Data\ASTM
ValidJP.ptm	For JP Validation	\(Folder which the IRsolution is installed)\Data\JP
ValidEP.ptm	For EP Validation	\(Folder which the IRsolution is installed)\Data\EP

7. Select the [File]-[Save] to save the each print template.
8. The setting is over.
9. Exit the IRsolution once and reactivate when the installation is completed. The following four menus are added to the [Measurement] menu when installation is correctly done.

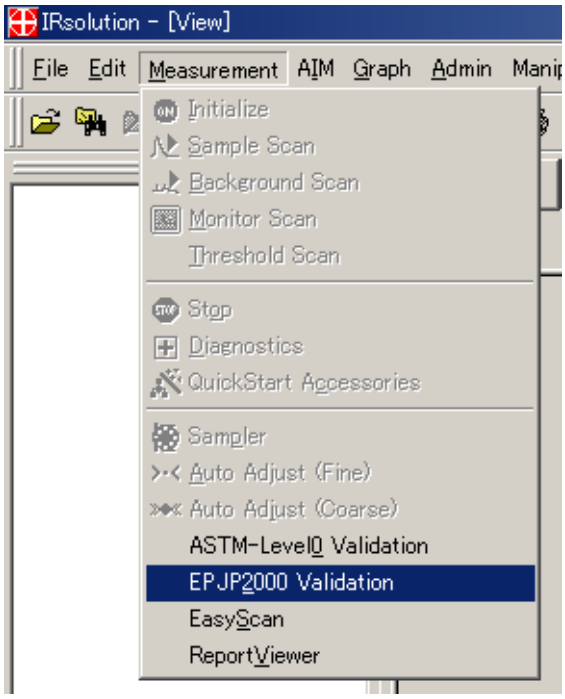


Fig. 2.3 Added Menus on the [Measurement] Menu

Chapter 3
Operation for the JP/EP Validation Program

The JP/EP Validation program complies with the description in 'General Test 23, Infrared Spectrophotometry' of the Fifteenth Edition of Japanese Pharmacopoeia, and "Absorption Spectrophotometry, Infrared" of the European Pharmacopoeia 5.0. For validation, use either the polystyrene film as standard accessory of the FTIR, the polystyrene film supplied by NIST, its secondary standard, or use the Beam Switching Kit. The Validation program measures the necessary spectra or uses saved spectra while running.

NOTE

1. Please purchase and use the Standard Reference Material of polystyrene film supplied by NIST or its secondary standard when traceable polystyrene film is needed.
2. Only FTIR-8300/8400/8400S, IRAffinity-1 and IRPrestige-21 are adapted to the European Pharmacopoeia 5.0.
3. Please input numbers by the normal-width figure on this program.

3.1 Preparation for Execution

3.1.1 Preparation

When the necessary spectra are measured while running the Validation program, the power switches for the FTIR, PC, and the printer should be turned on and the IRsolution should be activated permitting communication with the FTIR. Refer to the instruction manual of Shimadzu FTIR series for details.

It is necessary to install the optional Beam Switching Kit (P/N 206-70125-91/92) for the Automatic validation.

The following 3 spectra should be prepared to validate the FTIR with stored spectra.

- Power spectrum
- Polystyrene spectrum 1
- Polystyrene spectrum 2

Scan condition will be described in the later section.

3.1.2 Preparation of Printer

To print out the Validation report, confirm that a printer is turned on, cables are wired correctly, and the proper printer driver is installed. Refer to the instruction manuals for the printer and Windows for details.

3.1.3 Configuration of IRsolution

Confirm a configuration of IRsolution before running the Validation program.

(1) Procedure:

1. Open a spectrum.
2. Click the [View] tab.
3. Select the [Environment]-[Graph Preferences].
4. Select the [General] tab on the [2D Graphic Preferences] dialog box.
5. Set "10" for "Size" in "Character Format".
6. Click the [Style] tab.
7. Put check marks on the "Show peak borders" and the "Peaklabel on top of peak."

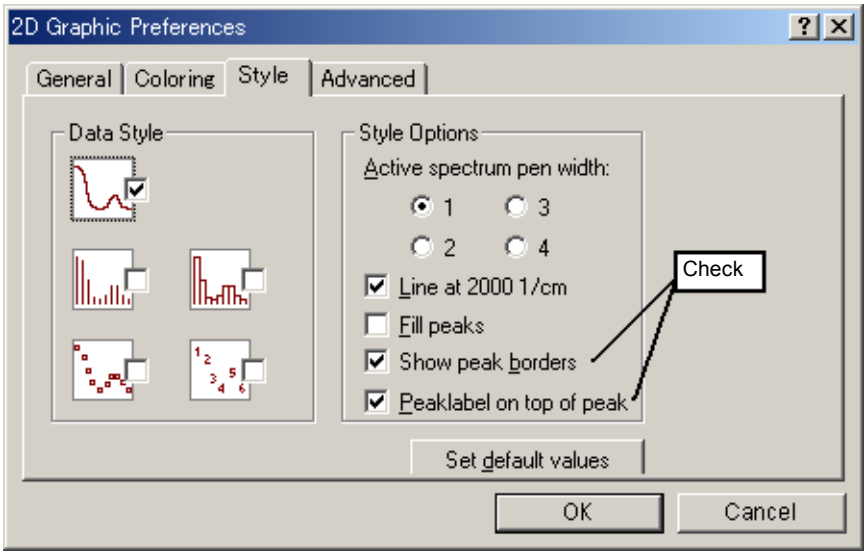


Fig. 3.1 [2D Graph Preferences] Dialog Box

8. Click the [OK] button.

(2) Display setting of background spectrum

1. Click the [Measure] tab.
2. Put a check mark on the "View background" on the screen.
3. Click the [Monitor] button to execute the monitor scan, and stop. (This operation is necessary to make the setting effective.)

3.2 Start the Validation Program

The program starts when the [Measurement]-[JP Validation] or [EP5.0 Validation] is selected.

The dialog box shown in Fig. 3.2 is displayed first to select the "**Instrument type**" among "IRPrestige/IRAffinity/FTIR-8400S" and Previous FTIR-8000series and FTIR-8200PC/8600PC. Inspection at 351 cm^{-1} is not done, when "FTIR-8000series" or "FTIR-8200PC/8600PC" is selected on the "Instrument" setting. Specifications of Power spectrum for each Instrument type are different.

NOTE

Only FTIR-8300/8400/8400S, IRAffinity-1 and IRPrestige-21 are adapted to the European Pharmacopoeia 5.0.

Fig. 3.2 [Select Instrument Type] Dialog Box

Select "IRPrestige/IRAffinity/FTIR-8400S", "FTIR-8000series" or "FTIR-8200PC/8600PC" of "Instrument Type" to be used by clicking the [▼] button, and then click the [OK] button. This dialog box is displayed when the Validation program is run for the first time. After that, it is not displayed, but the setting can be changed later.

The dialog box shown in Fig. 3.3 is displayed first to select the "**Report Language**" between Japanese and English. The messages in the displayed dialog box are all English.

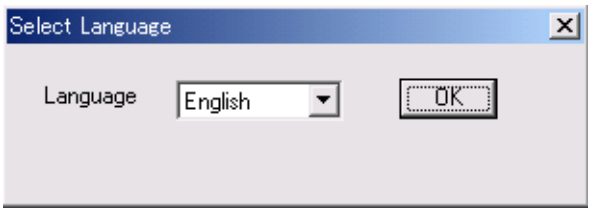


Fig. 3.3 [Select Language] Dialog Box

Select "Japanese" or "English" of report language suitable for your system by clicking the [▼] button, and then click the [OK] button. This dialog box is displayed when the Validation program is run for the first time. After that, it is not displayed, but the language can be changed later.

The following alert messages shown in Fig. 3.4 appear if other data files are displayed in the [View] tab.

Click [Yes] to close all displayed data. However, data will not be lost because the IRsolution includes an automatic saving function even if [No] is selected.

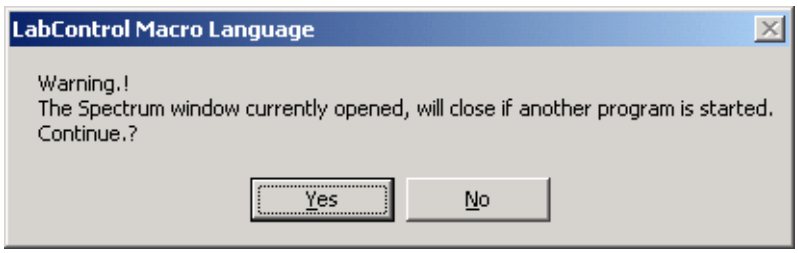


Fig. 3.4 Alert Message

When [Yes] is selected on the Fig. 3.3 or no data is opened on the [View] tab, the following dialog box shown in Fig. 3.5 is displayed.

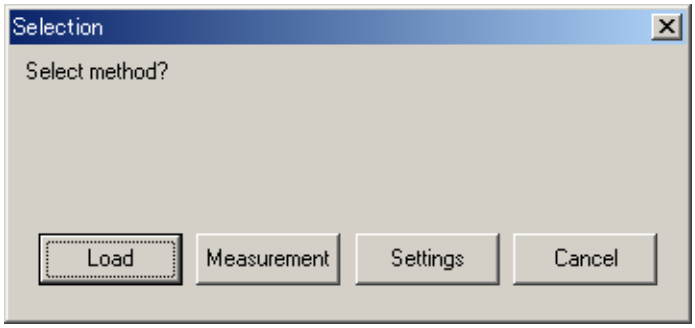


Fig. 3.5 [Selection] Dialog Box

Load	Run the Validation program with loading saved spectra. See section 3.5 for detail operation.
Measurement	Run the Validation program with scanning a polystyrene film.
Settings	Configures report language and the Validation mode (manual/auto).
Cancel	Cancels the program.

3.3 Settings

The following setting should be configured before running the Validation program.

- Instrument type to be used with is selected among "IRPrestige/IRAffinity/FTIR-8400S", Previous "FTIR-8000series" and "FTIR-8200PC/8600PC."
- Validation mode is selected between Manual validation and Automatic validation.
- Report language is selected between Japanese and English

To configure the setting, click the [Settings] button on the dialog box shown in Fig. 3.5. The dialog box shown in Fig. 3.6 is displayed.

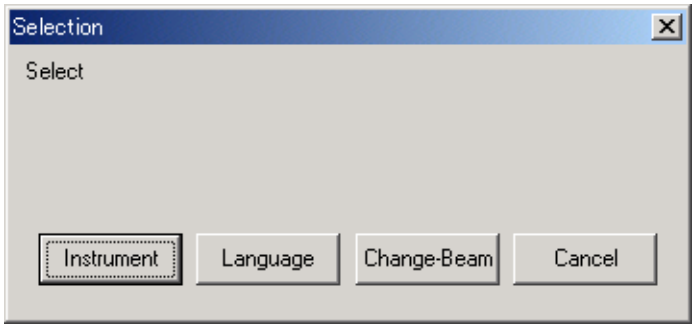


Fig. 3.6 Dialog Box to Configure Settings

A dialog box shown in Fig. 3.2 is displayed when the [Instrument] button is clicked. Select the Instrument type, then click the [OK] button.

A dialog box shown in Fig. 3.3 is displayed when the [Language] button is clicked. Select the language, then click the [OK] button. The Validation report will be printed in the selected language. The message on the dialog box is only in English.

When the [Change-Beam] button is clicked, a dialog box shown in Fig. 3.7 (to select manual or automatic validation) is displayed.

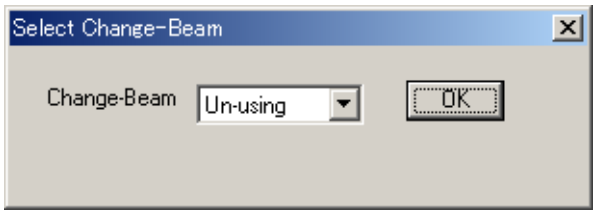


Fig. 3.7 Dialog Box to Select Manual or Automatic Validation

Select "Use" and click the [OK] button when the optional Beam Switching Kit (P/N 206-70125-91/92) is installed in Shimadzu FTIR series. When the kit is not installed, the "Un-using" option should be selected, and the [OK] button clicked.

The manual Validation program displays the message to set the polystyrene film in the sample compartment on the screen. The polystyrene film should be set into the sample holder.

The automatic Validation program inserts the polystyrene film installed on the Beam Switching Kit automatically and scans the necessary spectra.

3.4 Scan

When the [Measurement] button is clicked in Fig. 3.5, sample scanning is started. The [Parameter setting] dialog box shown in Fig. 3.8 is displayed. Input can be added to "Instrument", "Serial No.", "Temperature", "Sample name", "Relative Humidity", and "Inspected by." Printer output will display information provided by the user, columns left empty will appear blank.

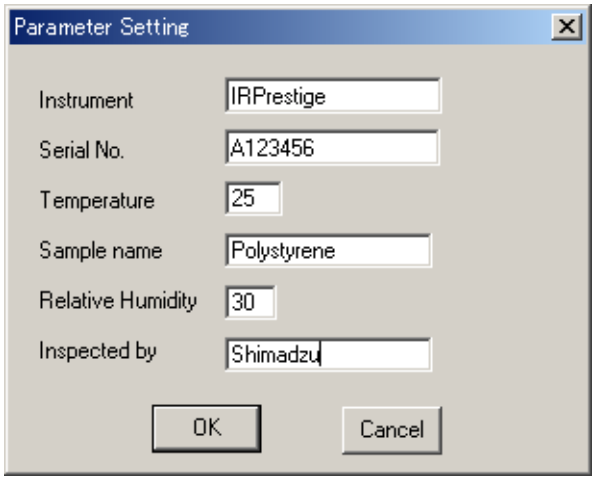


Fig. 3.8 [Parameter setting] Dialog Box

The message "Form of a power spectrum" shown in Fig. 3.9 is displayed. The power spectrum inspection starts when the [OK] button is clicked or automatically within 5 seconds.

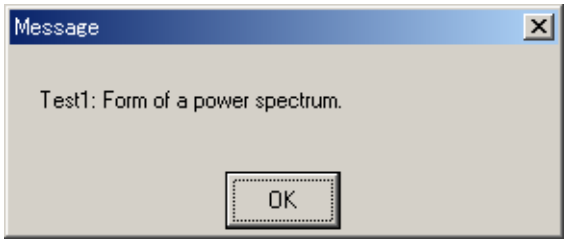


Fig. 3.9 Dialog Box to Start the Power Spectrum Inspection

The following message "Remove Sample from Sample Chamber" shown in Fig. 3.10 is displayed. Confirm that no sample is set on the sample compartment and then click the [OK] button. The scan parameter is sent to the interferometer and the measurement of the power spectrum starts. This power spectrum will also be the background of the following measurement.

Fig. 3.10 Message to Confirm the BKG Sample

After the power spectrum measurement, the polystyrene measurement starts. The message "Correctness of the resolution and wavenumber by polystyrene film." shown in Fig. 3.11 is displayed.

Fig. 3.11 Dialog Box to Start the Resolution and Wavenumber Accuracy Inspection

Click the [OK] button or wait 5 seconds for the message "Set Polystyrene Film into Sample Chamber." shown in Fig. 3.12 to be displayed. Set a polystyrene film, which is a standard accessory of FTIR or is supplied by NIST or its secondary standard for the manual Validation program, into the sample chamber. The Validation program automatically switches to the polystyrene file of the Beam Switching Kit. The polystyrene measurement is done twice.

Fig. 3.12 Message to Set a Polystyrene Film

The scan condition is as follows.

Resolution	2 cm^{-1}
No. of scans	45
Mirror speed	2.8 (low) mm/sec
Apodization	HAPP-GENZEL
Gain	Auto

Obtained spectra are stored into the following folders, respectively: for JP Validation, "c:\ProgramFiles\Shimadzu\IRsolution\Data\JP\[year]\[file name based on date/Time] such as 0207180743_1" and for EP Validation, "c:\ProgramFiles\Shimadzu\IRsolution\Data\EP\[year]\[file name based on date/Time] such as 0207180743_1". The obtained spectra are not overwritten when validation is done repeatedly. You can save the spectra by the [File]-[Save As] or using the Windows Explorer.

NOTE
The spectra obtained by IRsolution 1.30 or older are stored into a folder "c:\ProgramFiles\Shimadzu\IRsolution\Data\JP\[year]\[file name based on date/Time].

NOTE
To cancel the measurement scanning of the spectrum, the [Stop] button on the [Measure] tab is clicked, the [Stop Scanning] dialog box shown in Fig. 3.13 is displayed.

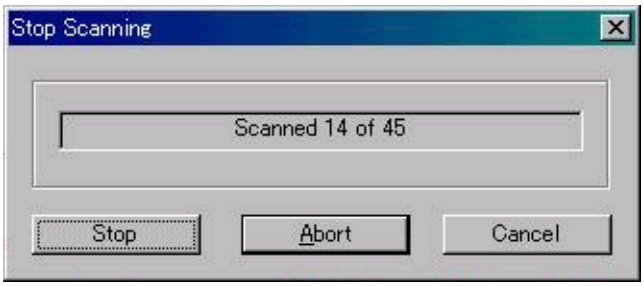


Fig. 3.13 [Stop Scanning] Dialog Box

Stop	Stops scanning and uses the measured spectrum.
Abort	Stops scanning and discards the measured spectrum.
Cancel	Continues the scanning.

When either [Stop] or [Abort] button is selected, the following dialog box is displayed.

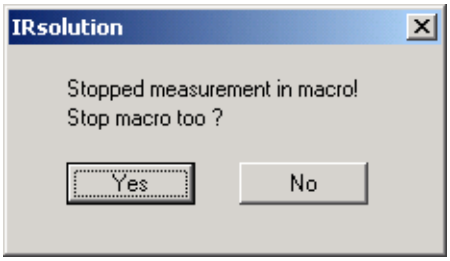


Fig. 3.14 [Stop Program] Dialog Box

The program ends when the [Yes] button is selected, and returns to the measurement mode. Proceed to the next step of this program when the [No] is selected.

3.5 Execution of Calculation

When the measurement is finished, calculation for the inspection is done automatically.

The Validation program inspects 5 tests. When all the tests are completed, the validation is passed. The following judgment dialog box shows "PASS" and a validation report is printed out.

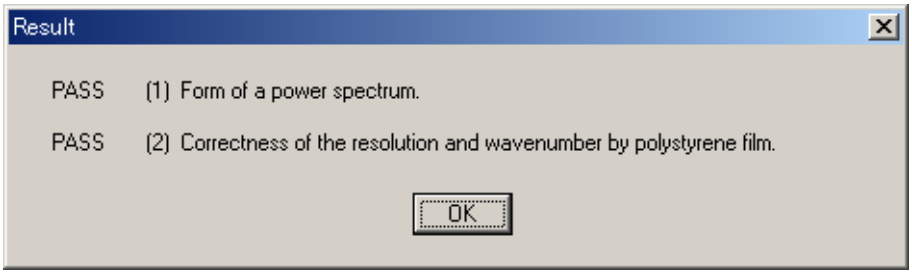


Fig. 3.15 [Result] Dialog Box with "PASS"

If one of the tests is not passed, the validation fails. The following judgment dialog box displays "FAIL" and a validation report is printed out.

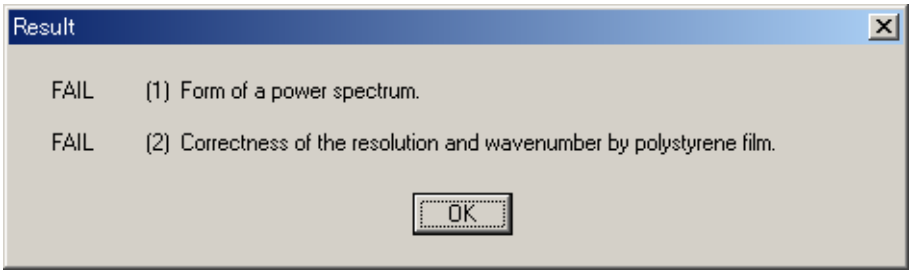


Fig. 3.16 [Result] Dialog Box with "FAIL"

This message closes when the [OK] button is clicked.

3.6 Validation Using Stored Data (JP/EP)

The Validation program can validate FTIR using stored spectra. Select the [Load] button on the [Selection] dialog box shown in Fig. 3.17. The [Parameter setting] dialog box shown in Fig. 3.17 is displayed.

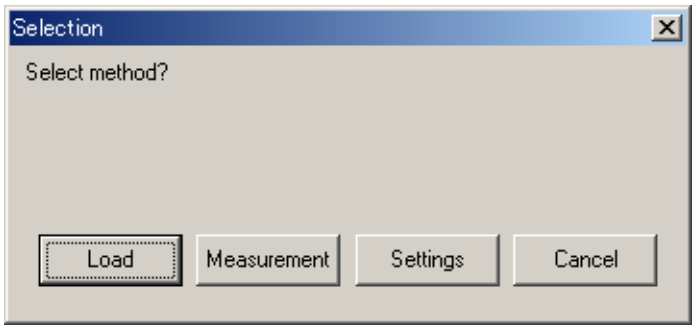


Fig. 3.17 Click [Load] button

Input can be added to "Instrument", "Serial No.", "Temperature", "Sample name", "Relative Humidity", and "Inspected by." Printer output will display information provided by the user, columns left empty will appear blank. Click the [OK] button after inputting the necessary information.

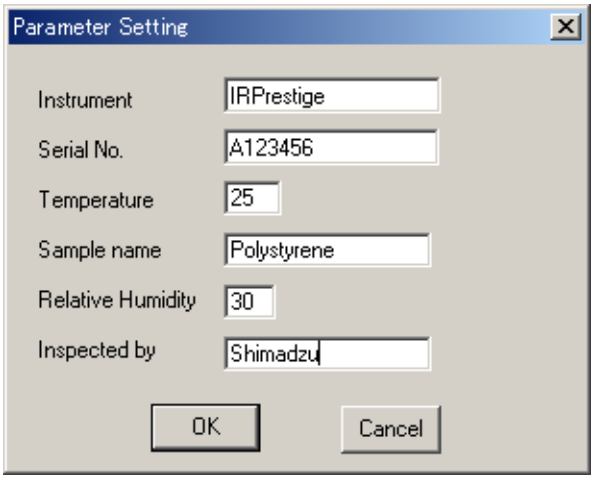


Fig. 3.18 [Parameter Setting] Dialog Box

The message "Form of a power spectrum" shown in Fig. 3.19 is displayed.

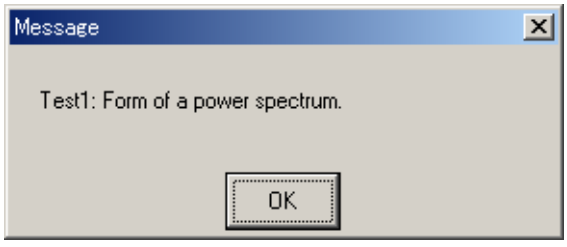


Fig. 3.19 Dialog Box to Start the Power Spectrum Inspection

The [Open Spectrum] dialog box shown in Fig. 3.20 is displayed when the [OK] button is clicked or automatically within 5 seconds. Select the necessary spectrum then click the [Save] button. The power spectrum is named with a file name based on created date and "_1."

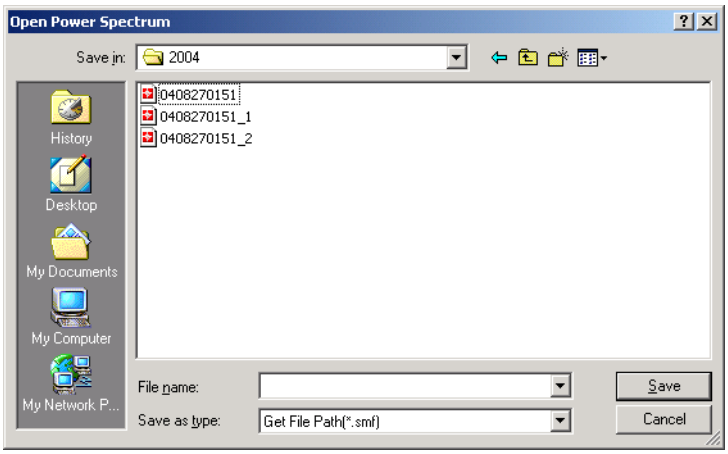


Fig. 3.20 [Open Spectrum] Dialog Box

After the power spectrum is loaded, the loading polystyrene spectra starts. The message "Correctness of the resolution and wavenumber by polystyrene film." shown in Fig. 3.21 is displayed.

Fig. 3.21 Dialog Box to Start the Resolution and Wavenumber Accuracy Inspection

The [Open Spectrum] dialog box shown in Fig. 3.22 is displayed again when the [OK] button is clicked or automatically within 5 seconds. Select the necessary polystyrene spectrum, then click the [Save] button. Repeat selecting a file twice. The polystyrene spectra are named with a file name based on created date and "_2" or "_3."

The validation result is automatically displayed and printed.

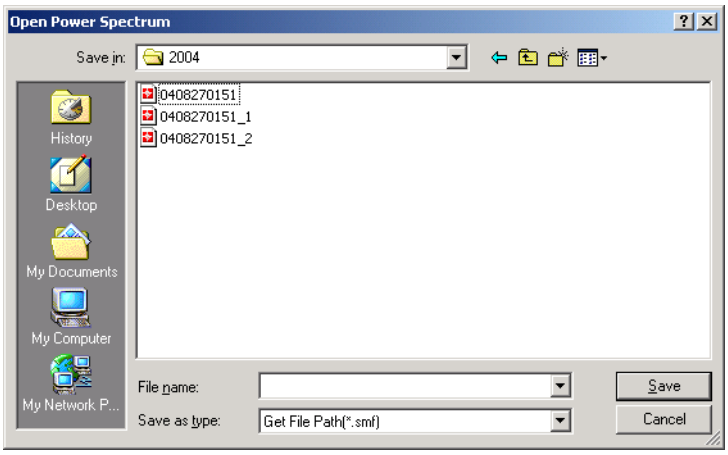
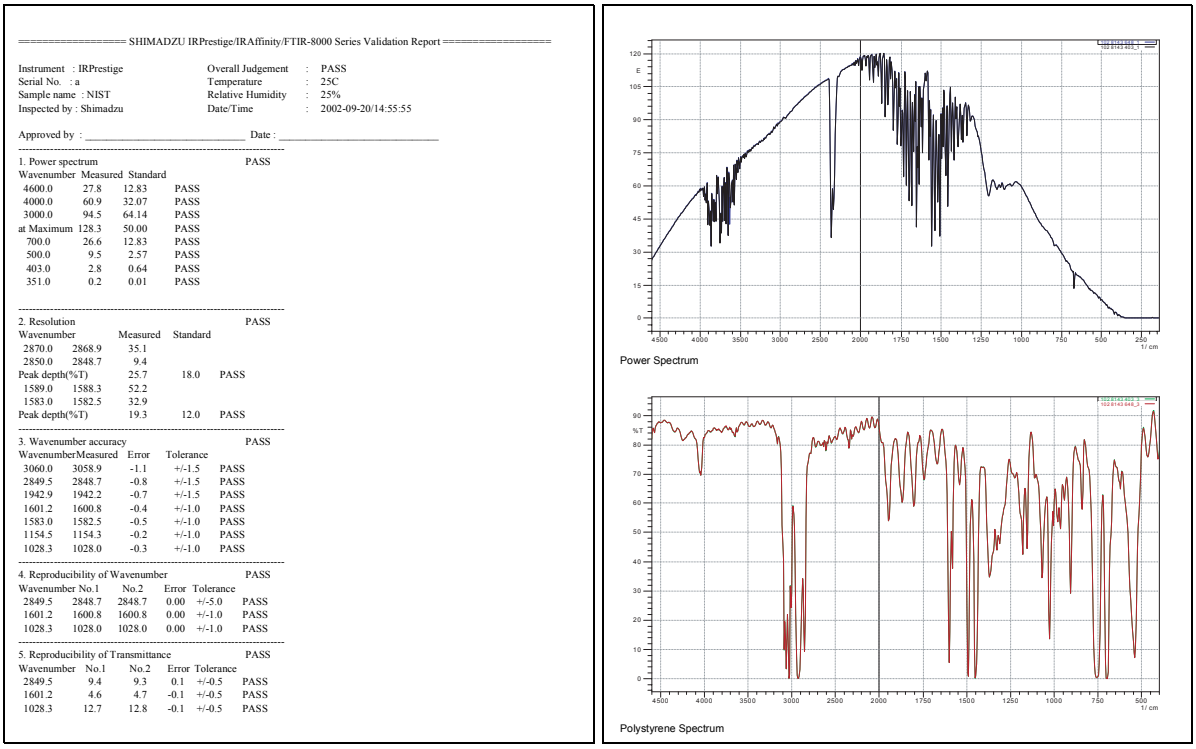


Fig. 3.22 [Open Spectrum] Dialog Box

3.7 Validation Report Example

An example of a validation report is shown below.



========== SHIMADZU IRPrestige/IRAffinity/FTIR-8000 Series Validation Report ==========

Instrument : IRPrestige
Serial No. : a
Sample name : NIST
Inspected by : Shimadzu

Overall Judgement : PASS
Temperature : 25C
Relative Humidity : 25%
Date/Time : 2002-09-20/14:55:55

Approved by : _____ Date : _____

1. Power spectrum PASS

Wavenumber	Measured	Standard	
4600.0	27.8	12.83	PASS
4000.0	60.9	32.07	PASS
3000.0	94.5	64.14	PASS
at Maximum	128.3	50.00	PASS
700.0	26.6	12.83	PASS
500.0	9.5	2.57	PASS
403.0	2.8	0.64	PASS
351.0	0.2	0.01	PASS

2. Resolution PASS

Wavenumber		Measured	Standard	
2870.0	2868.9	35.1		
2850.0	2848.7	9.4		
Peak depth(%T)		25.7	18.0	PASS
1589.0	1588.3	52.2		
1583.0	1582.5	32.9		
Peak depth(%T)		19.3	12.0	PASS

3. Wavenumber accuracy PASS

Wavenumber	Measured	Error	Tolerance	
3060.0	3058.9	-1.1	+/-1.5	PASS
2849.5	2848.7	-0.8	+/-1.5	PASS
1942.9	1942.2	-0.7	+/-1.5	PASS
1601.2	1600.8	-0.4	+/-1.0	PASS
1583.0	1582.5	-0.5	+/-1.0	PASS
1154.5	1154.3	-0.2	+/-1.0	PASS
1028.3	1028.0	-0.3	+/-1.0	PASS

4. Reproducibility of Wavenumber PASS

Wavenumber	No.1	No.2	Error	Tolerance	
2849.5	2848.7	2848.7	0.00	+/-5.0	PASS
1601.2	1600.8	1600.8	0.00	+/-1.0	PASS
1028.3	1028.0	1028.0	0.00	+/-1.0	PASS

5. Reproducibility of Transmittance PASS

Wavenumber	No.1	No.2	Error	Tolerance	
2849.5	9.4	9.3	0.1	+/-0.5	PASS
1601.2	4.6	4.7	-0.1	+/-0.5	PASS
1028.3	12.7	12.8	-0.1	+/-0.5	PASS

Fig. 3.23 Example of Validation Report

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Chapter 4
Operation for the ASTM Validation Program

"ASTM E1421-94 Level Zero" compares obtained data with stored reference data to detect changes.

A polystyrene film which is a standard accessory of the FTIR or is supplied from NIST is used as the sample. The same sample must be used in every inspection. If another sample is used, new reference data must be obtained. When an optical alignment is done for your FTIR, the reference data must be scanned again.

The reference file is saved in a specific folder. When the ASTM Validation program runs for the first time, it runs as a reference file measurement mode because there is no reference data. After that, the ASTM Validation program calculates data using the saved reference data. All of the measurement spectra are named by a file name based on the created date into a folder based on the created year. Files are save into "c:\Program Files\Shimadzu\IRsolution\Data\ASTM\[year]\[file name based on date/time]" such as 0207180743_1. The obtained spectra are not overwritten when validation is done repeatedly. You can save the spectra by the [File]-[Save As] or in Windows Explorer as a file name (The example:0207180743_1). And reference spectra are stored into "c:\Program Files\Shimadzu\IRsolution\Data\ASTM\REF."

R1.smf	Reference power spectrum1
R2.smf	Reference power spectrum2
R3.smf	Reference polystyrene spectrum

NOTE

- Please purchase and use the Standard Reference Material of polystyrene film supplied by NIST or its secondary standard when traceable polystyrene film is needed.
- ASTM validation must be performed after 2 hours and warming up of your FTIR.
- If ASTM validation fails, Auto-adjustment must be performed for optical alignment. After auto-adjustment, perform the JP and EP validation to check up the FTIR alignment. If the JP and EP validation fails, please contact your Shimadzu representatives. When the JP and EP validation passes, reference data for ASTM validation should be scanned again, and then ASTM validation should be run again.

4.1 Preparation for Execution

4.1.1 Preparation

When the necessary spectra are measured while running the Validation program, power switches for the FTIR, PC, and printer should be turned on and the IRsolution should be activated permitting communication with the FTIR. Refer to the instruction manual of Shimadzu FTIR series for details.

It is necessary to install the optional Bam Switching Kit (P/N 206-70125-91/92) for the Automatic validation.

The following 3 spectra should be prepared to validate the FTIR with stored spectra.

- Power spectrum1
- Power spectrum2
- Polystyrene spectrum

Scan conditions will be described in the later section.

4.1.2 Preparation of Printer

To print out the Validation report, confirm that the printer is turned on, cables are wired correctly, and the proper printer driver is installed. Refer to the instruction manuals for the printer and Windows for details.

4.1.3 Configuration of IRsolution

Confirm a configuration of IRsolution before running the Validation program.

(1) Procedure:

1. Open a spectrum.
2. Click the [View] tab.
3. Select the [Environment]-[Graph Preferences].
4. Select the [General] tab on the [2D Graphic Preferences] dialog box.
5. Set "10" for "Size" in "Character Format".
6. Click the [Style] tab.
7. Put check marks on the "Show peak borders" and the "Peaklabel on top of peak."

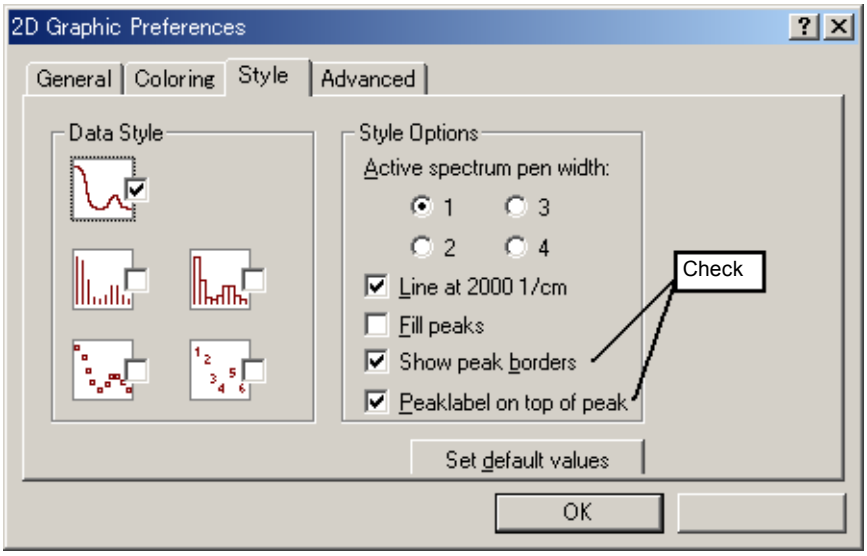


Fig. 4.1 [2D Graph Preferences] Dialog Box

8. Click the [OK] button.

(2) Display setting of background spectrum

1. Click the [Measure] tab.
2. Put a check mark on the "View background" on the screen.
3. Click the [Monitor] button to execute the monitor scan, and stop. (This operation is necessary to make the setting effective.)

4.2 Start the Validation Program

The program starts when the [Measurement]-[ASTM E1421-94 Level Zero Validation] is selected. The dialog box shown in Fig. 4.2 is displayed first to select the report language between Japanese and English. The messages on the displayed dialog box are all in English.

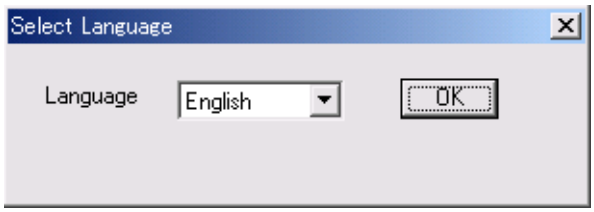


Fig. 4.2 [Select Language] Dialog Box

Select "Japanese" or "English" of report language to be suitable for your system by clicking the [▼] button, and then click the [OK] button. This dialog box is displayed when the Validation program is run for the first time. After that, it is not displayed, but the language can be changed later.

The following alert message shown in Fig. 4.2 appears if other data files are displayed in the [View] tab. Click [Yes] to close all displayed data. However, data will not be lost because the IRsolution includes an automatic saving function even if [No] is selected.

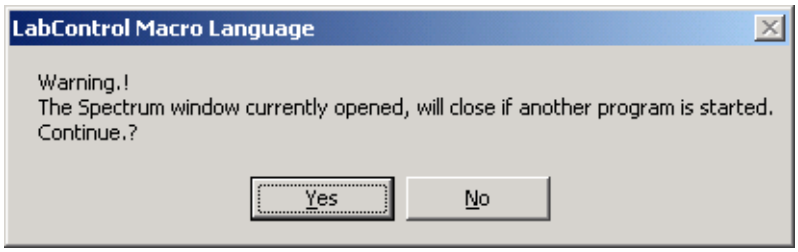


Fig. 4.3 Alert Message

When [Yes] is selected on the Fig. 4.3 or no data is opened on the [View] tab, the following dialog box shown in Fig. 4.4 is displayed.

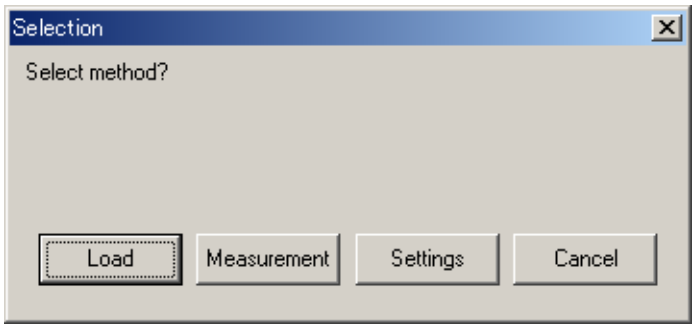


Fig. 4.4 [Selection] Dialog Box

Load	Run the Validation program with loading saved spectra. See section 4.6 for detail operation.
Measurement	Run the Validation program with scanning a polystyrene film.
Settings	Configures the report language and validation mode (manual/auto). And rescans reference spectra.
Cancel	Cancels the program.

4.3 Settings

The following setting should be configured before running the Validation program.

- Validation mode selected between Manual validation and Automatic validation.
- Report language selected between Japanese and English

To configure the setting, click the [Settings] button on the dialog box shown in Fig. 4.4. The dialog box shown in Fig. 4.5 is displayed.

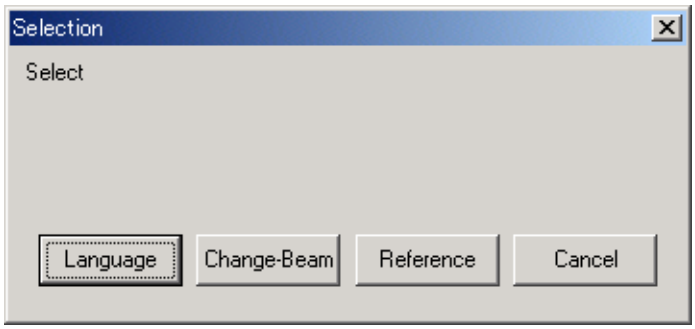


Fig. 4.5 Dialog Box to Configure Settings

A dialog box shown in Fig. 4.2 is displayed when the [Language] button is clicked. Select the language, then click the [OK] button. The Validation report will be printed in the selected language. The message in the dialog box is only in English.

When the [Change-Beam] button is clicked, a dialog box shown in Fig. 4.6 (to select manual or automatic validation) is displayed.

Fig. 4.6 Dialog Box to Select Manual or Automatic Validation

Select "Use" and click the [OK] button when the optional Beam Switching Kit (P/N 206-70125-91/92) is installed in Shimadzu FTIR series. When the kit is not installed, the "Un-using" option should be selected, and the [OK] button clicked.

The manual Validation program displays the message to set the polystyrene film in the sample compartment on the screen. The polystyrene film should be set into the sample holder.

The automatic Validation program inserts the polystyrene film installed on the Beam Switching Kit automatically and scans the necessary spectra.

When the [Reference] button is clicked, Rescanning of Reference spectra is started. Follow the prompts on the screen.

4.4 Scan

When the [Measurement] is clicked in Fig. 4.2, the sample scanning is started. The [Parameter setting] dialog box shown in Fig. 4.7 is displayed. Input can be added to "Instrument", "Serial No.", "Temperature", "Sample name", "Relative Humidity", and "Inspected by." Printer output will display information provided by the user, and columns left empty will appear blank.

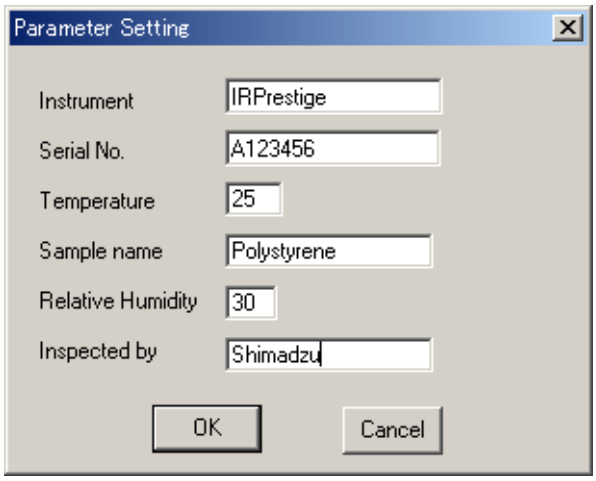


Fig. 4.7 [Parameter Setting] Dialog Box

When the [OK] button is clicked, the following message "Remove Sample from Sample Chamber" shown in Fig. 4.8 is displayed. Confirm that no sample is set on the sample compartment and then click the [OK] button. The scan parameter is sent to the interferometer and the measurement of the power spectrum starts. This power spectrum will also be the background of the following measurement.

Fig. 4.8 Message to Confirm the BKG Sample

After the power spectrum measurement, another power spectrum is scanned again.

After that, the polystyrene measurement starts. The message "Set Polystyrene Film into Sample Chamber." shown in Fig. 4.9 is displayed. Set a sample, and then click the [OK] button. The automatic Validation program automatically switches to the polystyrene file of the Beam Switching Kit.

Fig. 4.9 Message to Set a Polystyrene Film

The scan condition are as follows.

Resolution	4 cm^{-1}
No. of scans	20
Mirror speed	2.8 (low) mm/sec
Apodization	HAPP-GENZEL
Gain	Auto

Obtained spectra are stored into a folder "c:\Program Files\Shimadzu\IRsolution\Data\ASTM\[year]\[file name based on date/time]" such as 0207180743_1. The obtained spectra are not overwritten when validation is done repeatedly. You can save the spectra by the [File]-[Save As] or in Windows Explorer.

NOTE
To cancel the measurement during spectrum scanning, the [Stop] button on the [Measure] tab is clicked, the [Stop Scanning] dialog box shown in Fig. 4.10 is displayed.

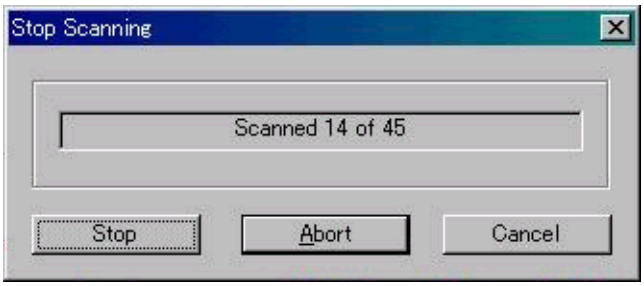


Fig. 4.10 [Stop Scanning] Dialog Box

Stop	Stops scanning and uses the measured spectrum.
Abort	Stops scanning and discards the measured spectrum.
Cancel	Continues the scanning.

When either [Stop] or [Abort] button is selected, the following dialog box is displayed.

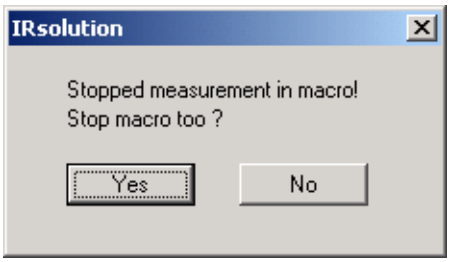


Fig. 4.11 [Stop Program] Dialog Box

This program ends once the [Yes] button is selected, and returns to the measurement mode. Proceed to the next step of this program when the [No] is selected.

4.5 Execution of Calculation

NOTE

When no reference data exists;

Only collection and saving reference spectra are done and calculation processing is not executed.

Execute this program again to make a validation report. When the measurement is finished, calculation for the inspection is automatically done.

The Validation program inspects 5 tests. When all the tests are completed, the validation is passed. The following judgment dialog box shows "PASS" and a Validation report is printed out.

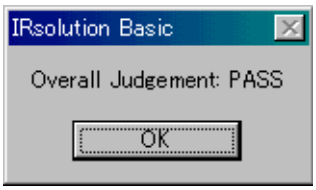


Fig. 4.12 [Judgment] Dialog Box with "PASS"

If one of the tests is not passed, the validation fails. The following judgment dialog box shows "FAIL" and a Validation report is printed out.

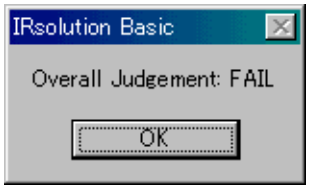


Fig. 4.13 [Judgment] Dialog Box with "FAIL"

This message box closes when the [OK] button is clicked.

4.6 Validation Using Stored Data (ASTM)

The Validation program can validate FTIR using stored spectra. Select the [Load] button on the [Selection] dialog box shown in Fig. 4.4. The [Parameter setting] dialog box shown in Fig. 4.14 is displayed.

Input can be added to "Instrument", "Serial No.", "Temperature", "Sample name", "Relative Humidity", and "Inspected by." Printer output will display information provided by the user, columns left empty will appear blank. Click the [OK] button after inputting necessary information.

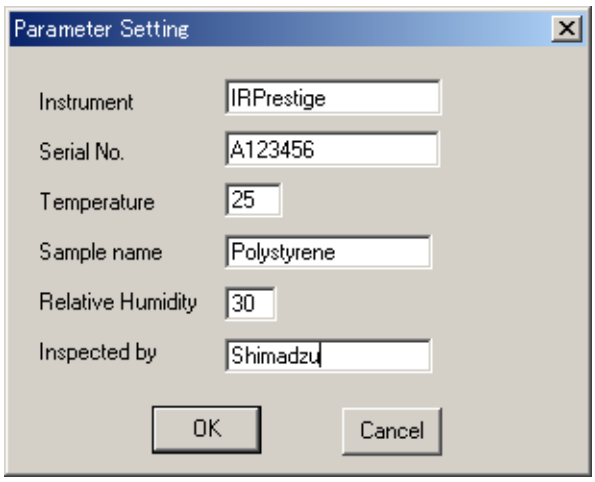


Fig. 4.14 [Parameter Setting] Dialog Box

The [Open Spectrum] dialog box shown in Fig. 4.15 is displayed when the [OK] button is clicked. Select the necessary spectrum then click the [Save] button. The power spectrum1 is named with a file name based on created date and "_1." The power spectrum2 is named with "_2" and the polystyrene spectrum is named with "_3."

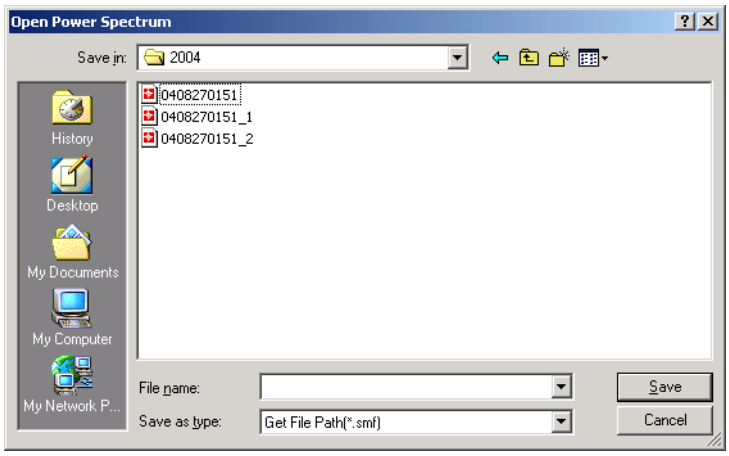


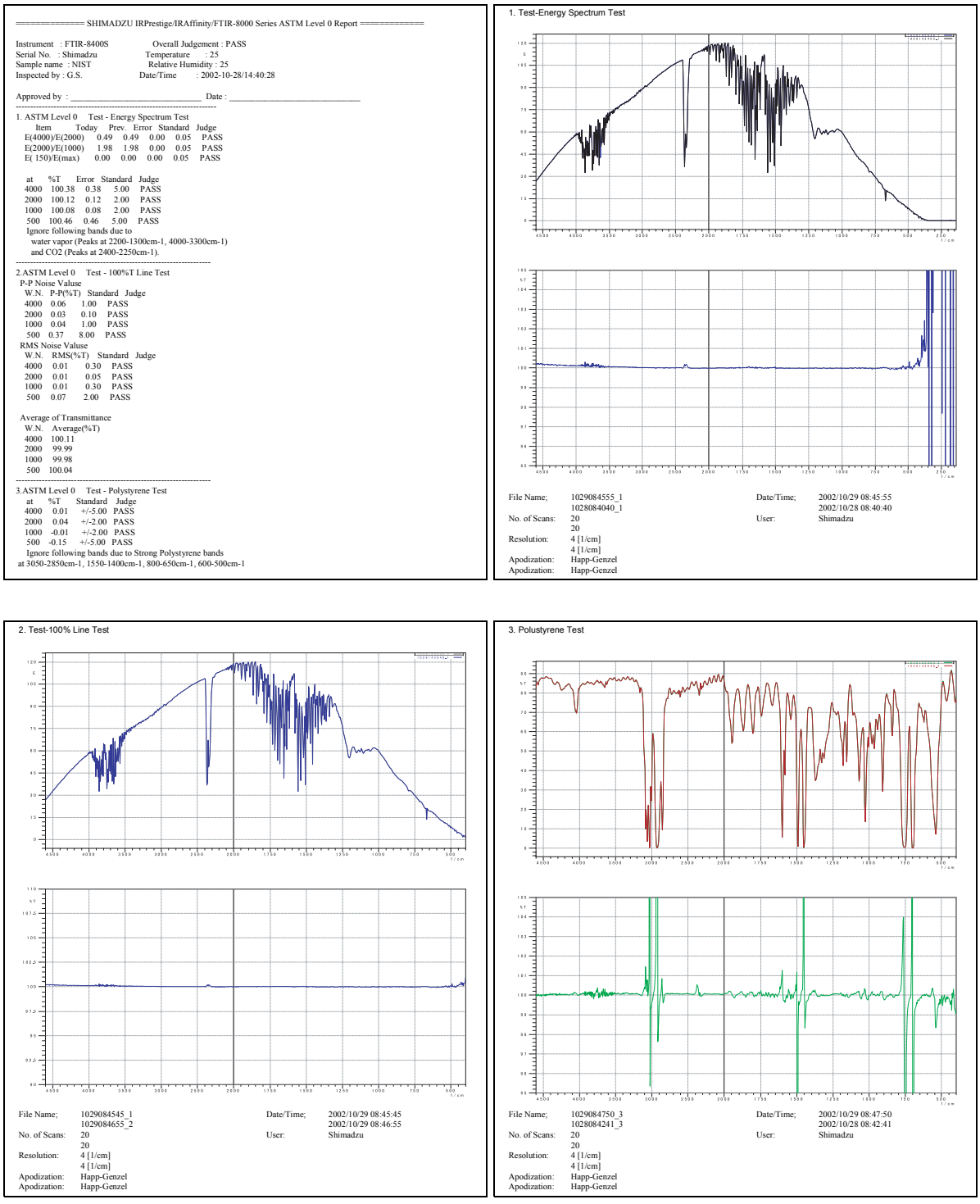
Fig. 4.15 [Open Spectrum] Dialog Box

Validation result is automatically displayed and printed out.

4.7 Validation Report Example

Here is an example of a validation report.

Scan parameters for two displayed spectra are printed out.



========== SHIMADZU IRPrestige/IRAffinity/FTIR-8000 Series ASTM Level 0 Report ==========

Instrument : FTIR-8400S Overall Judgement : PASS
Serial No. : Shimadzu Temperature : 25
Sample name : NIST Relative Humidity : 25
Inspected by : G.S. Date/Time : 2002-10-28/14:40:28

Approved by : _____ Date : _____

1. ASTM Level 0 Test - Energy Spectrum Test

Item	Today	Prev.	Error	Standard	Judge
E(4000)/E(2000)	0.49	0.49	0.00	0.05	PASS
E(2000)/E(1000)	1.98	1.98	0.00	0.05	PASS
E(150)/E(max)	0.00	0.00	0.00	0.05	PASS

at	%T	Error	Standard	Judge
4000	100.38	0.38	5.00	PASS
2000	100.12	0.12	2.00	PASS
1000	100.08	0.08	2.00	PASS
500	100.46	0.46	5.00	PASS

Ignore following bands due to
water vapor (Peaks at 2200-1300cm-1, 4000-3300cm-1)
and CO2 (Peaks at 2400-2250cm-1).

2.ASTM Level 0 Test - 100%T Line Test

P-P Noise Value

W.N.	P-P(%T)	Standard	Judge
4000	0.06	1.00	PASS
2000	0.03	0.10	PASS
1000	0.04	1.00	PASS
500	0.37	8.00	PASS

RMS Noise Valuse

W.N.	RMS(%T)	Standard	Judge
4000	0.01	0.30	PASS
2000	0.01	0.05	PASS
1000	0.01	0.30	PASS
500	0.07	2.00	PASS

Average of Transmittance

W.N.	Average(%T)
4000	100.11
2000	99.99
1000	99.98
500	100.04

3.ASTM Level 0 Test - Polystyrene Test

at	%T	Standard	Judge
4000	0.01	+/-5.00	PASS
2000	0.04	+/-2.00	PASS
1000	-0.01	+/-2.00	PASS
500	-0.15	+/-5.00	PASS

Ignore following bands due to Strong Polystyrene bands
at 3050-2850cm-1, 1550-1400cm-1, 800-650cm-1, 600-500cm-1

1. Test-Energy Spectrum Test

File Name; 1029084555_1
1028084040_1
No. of Scans: 20
20
Resolution: 4 [1/cm]
4 [1/cm]
Apodization: Happ-Genzel
Apodization: Happ-Genzel

Date/Time; 2002/10/29 08:45:55
2002/10/28 08:40:40
User: Shimadzu

2. Test-100% Line Test

File Name; 1029084545_1
1029084655_2
No. of Scans: 20
20
Resolution: 4 [1/cm]
4 [1/cm]
Apodization: Happ-Genzel
Apodization: Happ-Genzel

Date/Time; 2002/10/29 08:45:45
2002/10/29 08:46:55
User: Shimadzu

3. Polystyrene Test

File Name; 1029084750_3
1028084241_3
No. of Scans: 20
20
Resolution: 4 [1/cm]
4 [1/cm]
Apodization: Happ-Genzel
Apodization: Happ-Genzel

Date/Time; 2002/10/29 08:47:50
2002/10/28 08:42:41
User: Shimadzu

Fig. 4.16 Example of Validation Report

4.8 Troubleshooting

4.8.1 ASTM Validation Has Failed

If ASTM validation has failed, Auto-adjustment must be performed for optical alignment. After auto-adjustment, perform the JP and EP validation to check up the FTIR alignment. If the JP and EP validation has failed, please contact your Shimadzu representatives. When the JP and EP validation has passed, reference data for ASTM validation should be scanned again, then the ASTM validation should be run again.

4.8.2 Abnormal Spectra are on the Report

If abnormal results from Fig. 4.16 report are obtained, the reference spectra may be damaged for some reason.

Rescan the reference spectra with the [Reference] button referring to section 4.3.

Example of abnormal report)

- Page 2

  A Transmittance spectrum on upper side and a Power spectrum on the bottom side are printed, instead of Power spectra on the top and a 100%T spectrum on the bottom.

- Page 3

  A Transmittance spectrum on upper side and a Power spectrum on the bottom side are printed, instead of Power spectra on the top and a 100%T spectrum on the bottom.

- Page 4

  Two Transmittance spectra are printed, instead of Polystyrene spectra on the top and a 100%T spectrum on the bottom.

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Chapter 5
Operation of the Report Viewer

The inspection result is printed out and saved as a file on the hard disk. The report Viewer displays and prints out the stored Validation report. This section describes the operation of the Report Viewer.

5.1 Start up the Report Viewer

Select the [Measurement]-[Report Viewer] to start the report viewer. When the validation has not yet been done, the following message is displayed.

Fig. 5.1 Dialog Seen when Validation has not been Previously Performed.

The following window is displayed when there is a report.

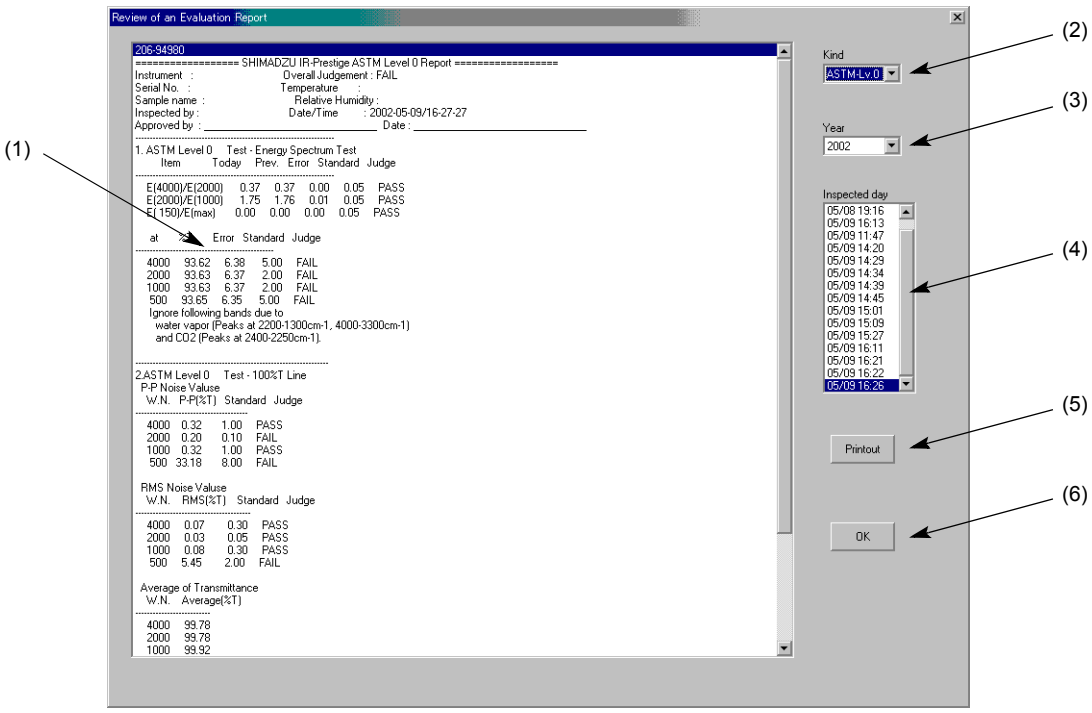

Fig. 5.2 Report Viewer Screen

5.2 Operation

Operation is done by using the button (1) to (6) in the Fig. 5.1. The explanation of each item is as follows.

(1) The content of the report is displayed here. When the whole of the screen is not displayed, the scrollbar displays the whole screen.

(2) Switches displayed report.

ASTM-Lv.0	Displays "ASTM E1421-94 Level Zero" validation report older than Version 1.10 program.
ASTM-Lv.0 [V1.10]	Displays "ASTM E1421-94 Level Zero" validation report in Version 1.10 or later.
Validation	Displays Reports created by JP and EP Validation with IRsolution 1.30 or older.
JP Validation	Displays Reports created by JP Validation, a standard accessory of IRsolution 1.40 or later.
EP Validation	Displays Reports created by EP Validation, a standard accessory of IRsolution 1.40 or later.

(3) The Validation report is stored in a different folder according to the year when the validation was done. Select the year of the data to be displayed.

(4) Displays the date and time when the report is created. Double-click one on the part to display it on the main screen (1).

(5) Prints the report displayed on the screen. The report assumes the A4 size form. Use papers of the A4 size.

(6) Exits the Report Viewer.

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Chapter 6
The Japanese Pharmacopoeia

The validation procedure complies with the description in 'General Test 23, Infrared Spectrophotometry' of the Fifteenth Edition of Japanese Pharmacopoeia. The software validates the equipment's performance by comparing the measured results to the standard values.

There are five parameters checked by this program: power spectrum, resolution, wavenumber accuracy, wavenumber reproducibility, and transmittance reproducibility.

The performance of FTIR is validated through comparison between the measurement result and standard values.

These characteristics are described in this chapter.

6.1 Power Spectrum

The intensity of the power spectrum is used to evaluate the basic performance of the FTIR. The power spectrum intensity is compared to a standard with specified wavenumbers. If the measured values are higher than the standard values at all the wavenumbers, the results are labeled "PASS."

The standard values are shown in the following table. They comprise the maximum value of power (shown in the center) and relational values of power, expressed as a percentage of the maximum value at a specified wavenumber. The standard values printed on the Validation report are calculated by the following specifications based on the actual maximum value of the power spectrum.

Wavenumber (cm^{-1})	**Standard value (IRPrestige-21/IRAffinity-1/ FTIR-8400S)**	**Standard value (FTIR-8000 series)**	**Standard value (FTIR-8201PC/8601PC)**
4600	10% or more of Max.	10% or more of Max.	14E or more
4000	25% or more of Max.	30% or more of Max.	30E or more
3000	50% or more of Max.	60% or more of Max.	40E or more
Maximum value	50.0 or more.	60.0 or more.	40E or more
700	10% or more of Max.	10% or more of Max.	8E or more
500	2% or more of Max.	2% or more of Max.	3E or more
403[1)]	0.5% or more of Max.	0.5% or more of Max.	0.4E or more
351[2)]	0.01% or more of Max.		

1) Water has a peak at 400 cm^{-1} and thus this program cannot calculate the correct value of the power spectrum at this value. The program uses the value 403 cm^{-1} as a baseline of the power spectrum instead of 400 cm^{-1}.

2) Inspection at 351 cm^{-1} is not done, when "FTIR-8000series" or "FTIR-8200PC/8600PC" is selected on the "Instrument" setting.

6.2 Resolution

The resolution is validated by measuring the absorption spectrum of polystyrene film with a thickness of approximately 0.04 mm. The measured absorption spectrum should have a transmittance (%T) difference of 18% or more between a minimum of approximately 2870 cm^{-1} and a maximum of approximately 2850 cm^{-1}. It should also have a transmittance (%T) difference of 12% or more between a minimum of approximately 1589 cm^{-1} and a maximum of approximately 1583 cm^{-1}. If the measured absorption spectrum has both transmittance differences higher than the standard value, the results are labeled "PASS."

6.3 Wavenumber Accuracy

Evaluating wavenumber accuracy is described in the Japanese Pharmacopoeia as follows:

The wavenumber scale is usually calibrated by using some of the following absorption bands of polystyrene film shown below. The numbers in parentheses indicate the allowable range for them.

3060.0 (+/- 1.5) cm^{-1}
2849.5 (+/- 1.5) cm^{-1}
1942.9 (+/- 1.5) cm^{-1}
1601.2 (+/- 1.0) cm^{-1}
1583.0 (+/- 1.0) cm^{-1}
1154.5 (+/- 1.0) cm^{-1}
1028.3 (+/- 1.0) cm^{-1}

This program uses seven wavenumber points for judging, and it obtains the peak wavenumbers from the measured spectrum of polystyrene film. The software then judges whether the values are within the allowable range. The program labels the results "PASS" if all the specified peak wavenumbers are within the allowable range.

6.4 Wavenumber Reproducibility

The Japanese Pharmacopoeia describes the wavenumber reproducibility as follows:

"The wavenumber reproducibility should satisfy 5 cm^{-1} around 3000 cm^{-1} of the polystyrene absorption wavenumber, 1 cm^{-1} around 1000 cm^{-1} when several points of polystyrene absorption, from 3000 cm^{-1} to 1000 cm^{-1}, are measured twice."

This program specifies three points to measure peak wavenumbers. It then obtains the actual peak wavenumbers at each point by measuring the polystyrene film twice. The software determines whether the differences between each of the two measurements are within the allowable range. The software labels the result "PASS" if all the specified peak wavenumbers are within the allowable range.

6.5 Transmittance Reproducibility

The Japanese Pharmacopoeia describes the transmittance reproducibility as follows:

"The transmittance reproducibility should satisfy 0.5%T when the several points of polystyrene absorption from 3000 cm^{-1} to 1000 cm^{-1} are measured twice."

This program specifies the peak wavenumber at three points and the transmittance at each point is measured twice. Then it is determined whether the differences between the two data are within the allowable range or not. If all the transmittance differences satisfy the allowable range, the results are labeled "PASS."

Chapter 7
The European Pharmacopoeia

The validation procedure complies with the description in "Absorption Spectrophotometry, Infrared" of the European Pharmacopoeia 5.0. The software validates the equipment's performance by comparing the measured results to the standard values.

There are five parameters checked by this program: power spectrum, resolving power (resolution), wavenumber accuracy, wavenumber reproducibility, and absorbance reproducibility.

The performance of FTIR is validated through comparison between the measurement result and standard values. The points differed from the EP4.0 are "Resolving Power (Resolution)", "Wavenumber Accuracy" and "Absorbance reproducibility".

These characteristics are described in this chapter.

NOTE

Only FTIR-8300/8400/8400S, IRAffinity-1 and IRPrestige-21 meet European Pharmacopoeia 5.0 regulation.

7.1 Power Spectrum

The intensity of the power spectrum is used to evaluate the basic performance of the FTIR. The power spectrum intensity is compared to a standard at the specified wavenumbers. If the measured values are higher than the standard values at all the wavenumbers, the results are labeled "PASS."

The standard values are shown in the following table. They comprise the maximum value of power (shown in the center) and relational values of power, expressed as a percentage of the maximum value at a specified wavenumber. The standard values printed on the Validation report are calculated by the following specifications based on the actual maximum value of the power spectrum.

Wavenumber (cm^{-1})	**Standard value (IRPrestige-21/IRAffinity-1/ FTIR-8400S)**	**Standard value (FTIR-8000 series)**
4600	10% or more of Max.	10% or more of Max.
4000	25% or more of Max.	30% or more of Max.
3000	50% or more of Max.	60% or more of Max.
Maximum value	50.0 or more.	60.0 or more.
700	10% or more of Max.	10% or more of Max.
500	2% or more of Max.	2% or more of Max.
403[1)]	0.5% or more of Max.	0.5% or more of Max.
351	0.01% or more of Max.	

1) Water has a peak at 400 cm^{-1} and thus this program cannot calculate the correct value of the power spectrum at this value. The program uses the value 403 cm^{-1} as a baseline of the power spectrum instead of 400 cm^{-1}

2) Inspection at 351 cm^{-1} is not done, when "FTIR-8000series" is selected on the "Instrument" setting.

7.2 Resolution

When the spectrum of polystyrene film about 35 μm thick is recorded, the difference between the absorbances at the absorption minimum at 2870 cm^{-1} and the absorption maximum at 2849.5 cm^{-1} is greater than 0.33. The difference between the absorbances at the absorption minimum at 1589 cm^{-1} and the absorption maximum at 1583 cm^{-1} is greater than 0.08. If the measured absorption spectrum has both absorbance differences higher than the standard value, the results are labeled "PASS."

7.3 Wavenumber Accuracy

Evaluating wavenumber accuracy is described in the European Pharmacopoeia 5.0 as follows:

"The wavenumber (cm^{-1}) scale may be verified using a polystyrene film, which has transmission minima (absorbance maxima) at the wavenumbers (in cm^{-1})."

3060.0 (+/- 1.0) cm^{-1}
2849.5 (+/- 1.0) cm^{-1}
1942.9 (+/- 1.0) cm^{-1}
1601.2 (+/- 1.0) cm^{-1}
1583.0 (+/- 1.0) cm^{-1}
1154.5 (+/- 1.0) cm^{-1}
1028.3 (+/- 1.0) cm^{-1}

This program uses seven wavenumber points for judging, and it obtains the peak wavenumbers from the measured spectrum of polystyrene film. The software then judges whether the values are within the allowable range. The program labels the results "PASS" if all the specified peak wavenumbers are within the allowable range.

7.4 Wavenumber Reproducibility

European Pharmacopoeia 5.0 does not have this test, but this validation program inspects based on the Japanese Pharmacopoeia.

The Japanese Pharmacopoeia describes the wavenumber reproducibility as follows:

"The wavenumber reproducibility should satisfy 5 cm^{-1} around 3000 cm^{-1} of the polystyrene absorption wavenumber, 1 cm^{-1} around 1000 cm^{-1} when several points of polystyrene absorption, from 3000 cm^{-1} to 1000 cm^{-1}, are measured twice."

This program specifies three points to measure peak wavenumbers. It then obtains the actual peak wavenumbers at each point by measuring the polystyrene film twice. The software determines whether the differences between each of the two measurements are within the allowable range. The software labels the result "PASS" if all the specified peak wavenumbers are within the allowable range.

7.5 Absorbance Reproducibility

European Pharmacopoeia 5.0 does not have this test, but this validation program inspects based on the Japanese Pharmacopoeia. Tolerance is specified of absorbance which is nearly equivalent to 0.5% transmittance at the wavenumber.

The Japanese Pharmacopoeia describes the transmittance reproducibility as follows:

"The transmittance reproducibility should satisfy 0.5%T when the several points of polystyrene absorption from 3000 cm^{-1} to 1000 cm^{-1} are measured twice."

This program specifies the peak wavenumber at three points and the absorbance at each point is measured twice. Then it is determined whether the differences between the two data are within the allowable range or not. If all the absorbance differences satisfy the allowable range, the results are labeled "PASS."

Chapter 8
"ASTM E1421-94 Level Zero"

This software complies with the description in the "ASTM (American Society for Testing and Materials) E1421-94 Level Zero". There are three parameters checked by this program: Energy Spectrum Test, One Hundred Percent Line Test, and Polystyrene Test.

These characteristics are described in this chapter.

Brief explanation of the ASTM E1421-94 Level Zero

This is a simple test done within a couple of minutes. ASTM E1421-94 Level Zero compares obtained data with stored reference data obtained after optical alignment to detect changes. It is not the one to do a quantitative strict evaluation.

The scan parameters are 4 cm^{-1} resolution, and about 30 seconds accumulation. It is necessary to record other scan parameters.

Reference1	Reference power spectrum
Reference2	Reference polystyrene spectrum
Spectrum1	Blank power spectrum1
Spectrum2	Blank power spectrum2 measured after Spectrum1
Spectrum3	Polystyrene spectrum with Spectrum1 as BKG

8.1 Energy Spectrum Test

The Energy Spectrum Test compares the power spectrum to evaluate any changes in the long term.
The following results were reported.

(1) Spectrum1 and Reference1 overlaid.

(2) 100%T line spectrum calculated by (Spectrum1)/(Reference1) in 100 +/- 10% scale.

(3) Energy Ratios listed up on following table. When the energy ratios are within the standard, result is "PASS."

Energy Ratio

$R_{4000/2000}=E_{4000}/E_{2000}$	+/- 0.3
$R_{2000/1000}=E_{2000}/E_{1000}$	+/- 0.3
$R_{nonphysical}=E_{150}E_{max}$	+/- 0.3

Error standard for 100%T line

4000 cm^{-1}	+/- 30
2000 cm^{-1}	+/- 30
1000 cm^{-1}	+/- 30
500 cm^{-1}	+/- 30

8.2 One Hundred Percent Line Test

One Hundred Percent Line Test calculates 100%T line (Spectrum2/Spectrum1) to evaluate any short-term changes.

The following results were reported.

(1) Spectrum1 and Spectrum2 overlaid.

(2) 100%T line spectrum calculated of (Spectrum2)/(Spectrum1) in 100 +/- 10% scale.

(3) RMS or Peak-to-Peak noise levels of the 100%T line spectrum at 100 cm^{-1} range centered at 4000, 2000, 1000, 500 cm^{-1}. When the noise levels are within the standard, "PASS" results.

Peak-to-Peak noise level standard

4000 cm^{-1}	1.00
2000 cm^{-1}	0.10
1000 cm^{-1}	1.00
500 cm^{-1}	8.00

RMS noise level standard

4000 cm^{-1}	0.50
2000 cm^{-1}	0.05
1000 cm^{-1}	0.50
500 cm^{-1}	4.00

8.3 Polystyrene Test

Polystyrene Test compares the polystyrene spectrum (Reference2 and Spectrum3) to evaluate any long term changes.

The following results were reported.

(1) Spectrum3 and Reference2 overlaid.

(2) Subtracted spectrum between Spectrum3 and Reference2 in100 +/- 10%T scale*).

(3) When the differences are within the standard, "PASS" results.

Standard differences of the subtracted data.

4000 cm^{-1}	10
2000 cm^{-1}	10
1000 cm^{-1}	10
500 cm^{-1}	10

* The subtracted spectrum should be printed in +/- 1%T scale in section 7.3.1. on the ASTM E1421-94. But this program prints out the subtracted spectrum in +/- 10%, because the maximum of standard in the program is +/- 10%.

Record of Revision

Date	Revision	Changed Page	Description
12/2006	A	New	New
08/2007	B	Add: Descriptions for IRAffinity-1	