

FTIR

TALK LETTER

Vol. 23



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LabSolutions IR Report Template Creation Procedures

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In the infrared spectrophotometer control workstation LabSolutions IR, more functions have been added and improved in comparison to previous control software IRsolution, making it easier to use. One of these is the report template function. This function allows the user to freely edit the report output items and the layout. This article will introduce the report template creation procedures and registration procedures.

1. Report Templates

The LabSolutions IR report template function is used to create templates for arranging and printing measurement data graphs, parameters, spectral search results, and other information in the [Report] window for printing output results. With LabSolutions IR, numerous report templates are provided as standard. However, users can also create report templates to suit their objectives and in formats that are easier to understand. The templates created can be saved as report template files. A particular template can be specified for printing, and printing can be linked with the LabSolutions IR windows.

2. Improvements from IRsolution

The LabSolutions IR report template function reflects the following improvements in comparison to IRsolution.

The printing results can be edited with the images as is. (See Fig. 1.)

When arranging items in the [Edit Printform] window with IRsolution, only the frame is displayed, not the actual graph (Fig. 1 (a) View|Graph), and the character string "\$ (IR Spectrum|Created)" is displayed in place of the date and time item, rather than the content actually printed. In contrast, with LabSolutions IR, the actual graph waveform and the actual date and time are displayed. As a result, it takes less time to check the layout and make fine adjustments to the item arrangement.

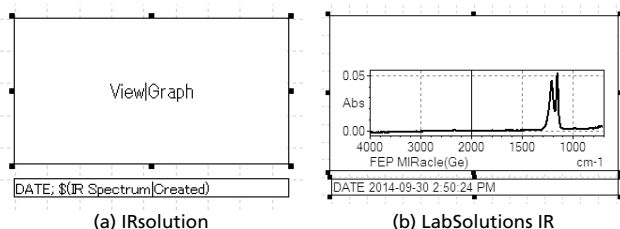


Fig. 1 The [Edit Printform] Window Display

3. Items Can Be Edited in the Report Template Editing Window

In the report template editing window, the printing layout can be edited by dragging graphs, peak tables, and other required items into the [Report] window. (See Fig. 2.) Items can be consolidated one by one, or you can click the \oplus icon to the left of an item to display an expanded view of the items included therein.

In addition to changing the position and size of items in the [Report] window, you can configure the characteristic settings for each item in the [Property] window. For example, for the spectral graph item, you can show or hide the grid, and specify the items for the X-axis range.

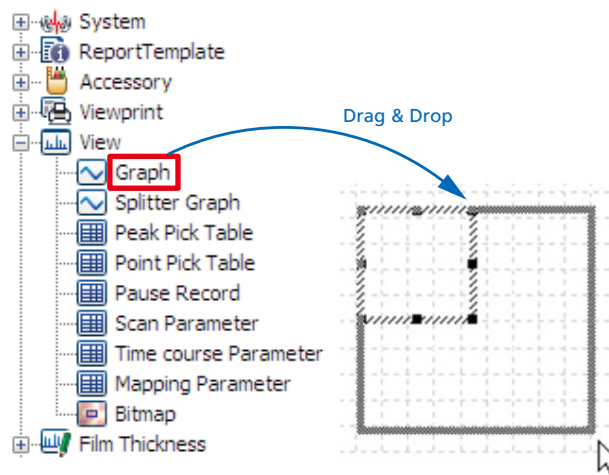


Fig. 2 Adding Items

4. Basic Report Template Creation

This section describes the procedures for creating a report template for printing peak detection results. As prerequisites, the spectral data file must already be opened, and peak detection must have been implemented.

First, to edit the report template, open the [Edit Printform] window. To display the [Edit Printform] window, click the [Edit Printform] tab on the main

toolbar in the LabSolutions IR postrun analysis program or the measurement program. Next, drag and drop the items to output in the report from the [Item] window to the [Report] window. Here, I have selected (1) [Date&Time] and (2) [User Name] from the [System] category, (3) [Graph], (4) [Peak Pick Table], and (5) [Scan Parameter] from the [View] category, and (6) [Filename] from the [Property] – [Spectrum] category. (See Fig. 3.)

When an item is activated by clicking it, the configurable settings are displayed in the [Property] window. The item properties include the [Data

Source] setting. By default, the spectral data on the active tab, i.e. the tab that is active in the display window, is set as the source.

For graph display items as in (3), you can show or hide the grid, or set the X-axis range in the [Property] window. The graph line color and thickness settings are reflected by those in the display window, so to edit them, right-click the graph in the display window, and on the displayed menu, click [Graph Preferences]. Note that to acquire a graph as an image, click [Copy] on the [Edit] menu to copy the graph image to the clipboard.

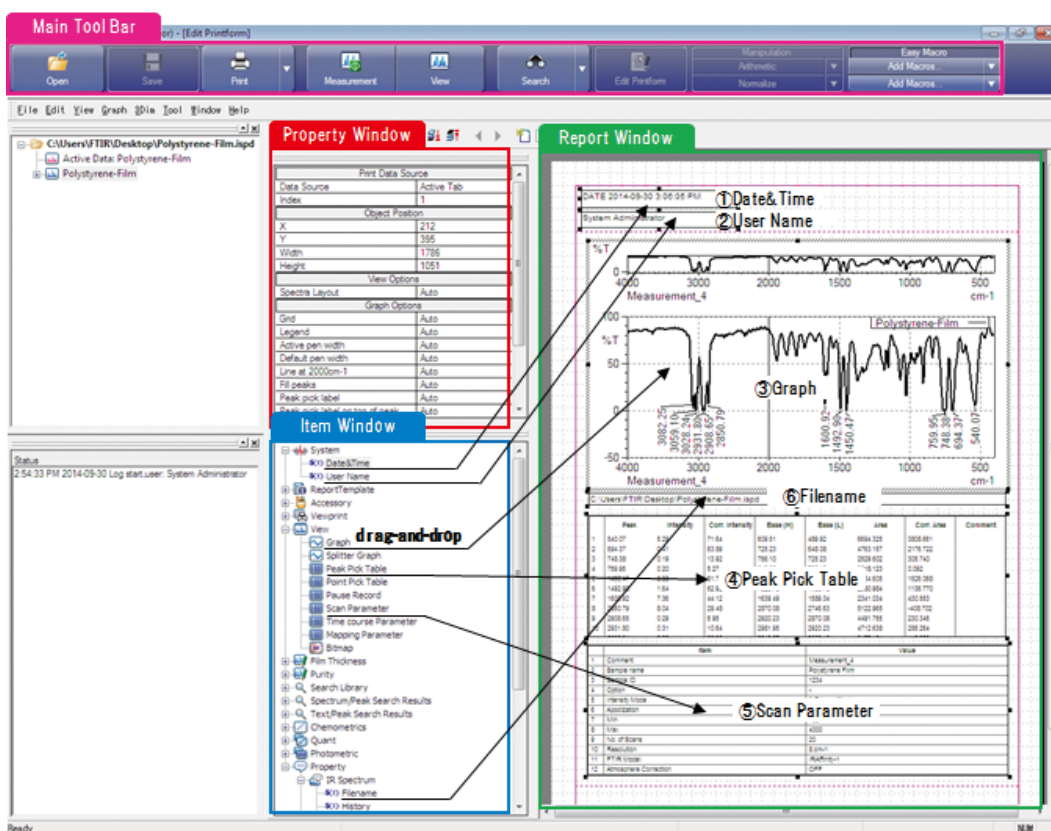


Fig. 3 The [Edit Printform] Window

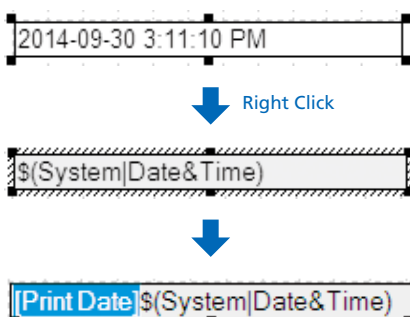


Fig. 4 Adding Text

For text output items such as (1), (2), and (6), right-click the item, and click [Edit Mode] on the menu to add text as you wish. (See Fig. 4.) For example, with the item (1) [Date&Time], only the date and time are output, so you can add the text "Print Date" before the date and time to clarify the meaning. (See Fig. 4.) You can also edit the font type, the style, or the size by clicking [Edit Mode].

With the item (4) [Peak Pick Table], there is no item in the [Property] window specifying the peak No. to be output in the report. To output a specific peak only, select the unnecessary rows in the peak table in the [View] window. Click [Delete Peak] on the right-click menu. The rows deleted from the peak table will not be output in the report.

In terms of scan parameters, you can either select the [Scan Parameter] item from the [View] category as in (5), or select individual items from the scan parameters in the [Property] – [Scan Parameter] category. In the former case, scan parameters can be added in batch to a report, but there are many such scan parameters, so to select only the required items, click the button to the right of [Scan Parameter] in the [Data Options] area. A window will then be displayed for setting the items to print.

Only items with the corresponding checkbox selected in the [Print] column in this window will be output in the report. (See Fig. 5.) In the latter case, you can add the required parameters one at a time as individual items in the report.

When you have determined the item content and the layout, click [Save as] on the [File] menu. To print as is, click [Print Preview] on the [File] menu. Confirm the print preview, and then click [Print].

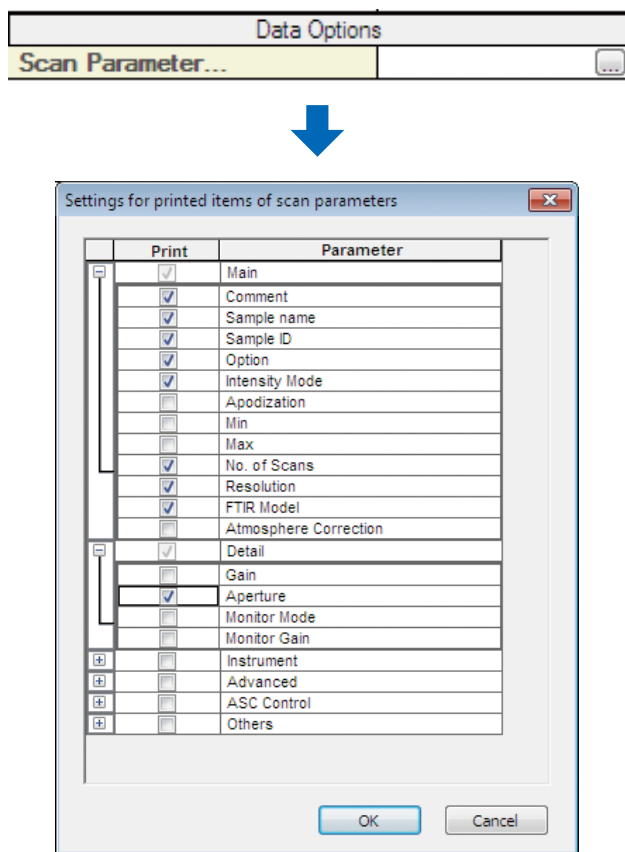


Fig. 5 Scan Parameters to Print

5. Registering Report Templates

To print a saved report template file, click [Print with Template] on the [File] menu. Also, by clicking [Print Preferences] on the [Tool] menu, it is possible to link a default report template file to printable windows. For example, in the default case, the report template "data_p.irtm" is allotted to the display window, but by changing this, you can avoid the trouble of clicking [Print with Template] each time.

6. Changing the Number of Decimal Places

With LabSolutions IR, you can change the number of decimal places for measurement data for each item. (See Fig. 6.)

The procedure is as follows.

- (1) If LabSolutions IR is running, exit it.
- (2) Run LabSolutions Manager. (Log in to LabSolutions with administrator rights.)
- (3) Click [Administration], point to [System Settings], and select [Data Proc. Settings].
- (4) Set the number of decimal places for each data item.
- (5) Exit LabSolutions Manager.

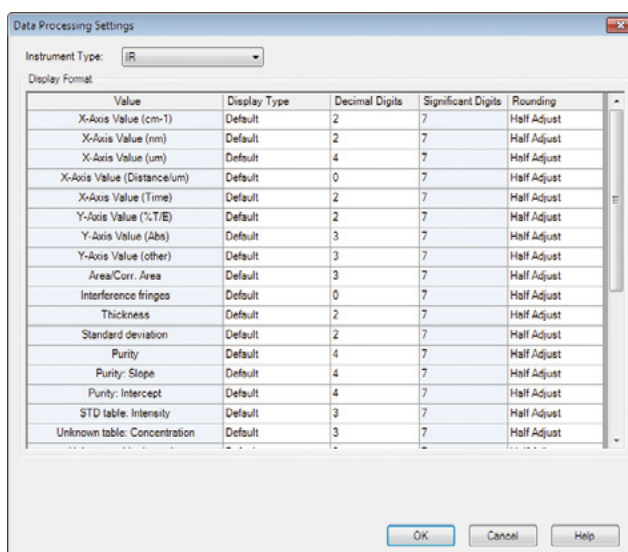


Fig. 6 The Window for Changing the Number of Decimal Places

7. Summary

The report templates introduced here can be created, edited, and registered with a series of intuitive operations, considerably improving the ease of use. Please use this function to create report templates to suit your analysis objectives and measurement data operations, thereby enhancing productivity.

Utilizing User Libraries

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Libraries (databases) are indispensable for spectral searches. They are broadly classified into two types: commercially available libraries and user libraries. Commercially available libraries (including those provided as standard with instruments) contain information on general substances and compounds. In contrast, user libraries are constructed by users themselves, so they can contain arbitrary IR spectra and information.

1. Introduction

Vol. 22, an earlier edition, introduced the use of difference spectra as an approach to spectral analysis. It was noted that difference spectra are effective for comparing small peak differences and for identifying contaminants. However, the use of user libraries is also an effective approach to spectral analysis. In this article, I will introduce their features and look at some actual examples.

2. Features of User Libraries and Commercially Available Libraries

IR spectra are specific to each substance, and are suitable for the qualitative analysis of organic substances. In this case, libraries are effective, but it should be noted that commercially available libraries differ by measurement method and category, and there are numerous options. In contrast, users can also create user libraries themselves.

For example, IR spectral peak intensities and wave number position are well known to differ depending on the measurement method. Specular reflectance spectra can be converted into general absorption spectra using Kramers–Kronig analysis. Diffuse reflectance spectra can be similarly converted using Kubelka–Munk conversion, and ATR spectra can be converted using ATR correction. If commercially available libraries are used, these corrections and conversions will be necessary if there is a difference between the measurement method in the library, and the measurement method for the IR spectra subject to search. However, if a user library is used, searches can proceed without corrections or conversions if the measurement method for the IR spectra subject to search is matched to the measurement method for the registered IR spectra.

In addition, in spectral searches, a score (degree of similarity) is calculated from the shape of the IR spectrum as a whole. As a result, making determinations from the score alone will be difficult if the IR spectral differences are small. With commercially available libraries, it is not easy to distinguish the difference between low-density polyethylene and high-density polyethylene, or between ethylene-propylene block and random copolymers, because the difference between scores is small. However, with user libraries, even if the difference between scores is not large, detailed identification and qualification can be performed by preregistering the library with relevant peak information.

When creating user libraries, sample acquisition and measurement conditions are indispensable, so time and effort are needed. However, when registering a sample serving as a foreign substance, for example, adding detailed information on the target substance including the sample name, part No., and place of use, may prove useful for investigations to determine the cause of foreign substance contamination.

In contrast, an advantage of commercially available libraries is that a large amount of data can be obtained without the effort involved in measuring them. However, they must be purchased, and only contain information on typical substances.

Also, most such libraries are associated with a particular brand, so analysis can be difficult if, for example, the spectral search involves a mixed spectrum. Also, in foreign substance analysis, even if qualitative and identification information is obtained, separate investigation will be required to determine the cause (i.e. the origin of the foreign substance).

A comparison of user libraries and commercially available libraries (advantages and disadvantages) is summarized in Table 1.

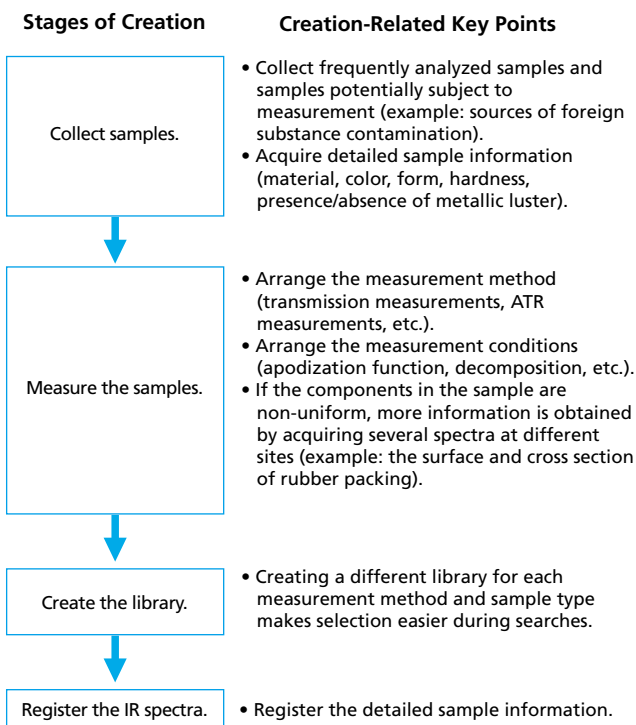
Table 1 Comparison of User Libraries and Commercially Available Libraries

	User Libraries	Commercially Available Libraries
Advantages	Inexpensive Any data and information can be registered. The cause of foreign substance contamination can be directly determined.	Large amounts of data can be obtained. No measurements are required.
Disadvantages	Samples must be acquired and measured. →Effort is involved. Not many substances are registered.	Expensive Only typical substances are registered. Only substance names and other typical information are contained. →The causes of foreign substance contamination will remain unknown.

3. Procedures and Key Points When Creating User Libraries

The procedures for creating a user library are summarized in Table 2.

Table 2 Procedures for Creating User Libraries



Key points related to the creation of user libraries are noted above. To improve the accuracy of spectral searches, however, the quality of the IR spectra added to the library is also important. Particularly for transmission measurements, be careful

that the maximum peak in absorbance does not exceed the range for which linear data can be obtained from the detector (normally an absorbance between about 0 and 1). If this range is exceeded, the peak will plateau, and the peak height ratio will end up changing. Also, atmospheric correction is required for carbon dioxide, water vapor and other substances greatly affected by the air. During library registration, the values will be standardized automatically using the maximum value, so baseline correction is not required. However, but pay attention to the issues noted above to ensure that you create a high-quality library.

It is also recommended that you incorporate as much sample information as possible into user libraries as comments.

4. Introduction to a Library for Foreign Substances in the Water Supply

This section describes a library for foreign substances in the water supply as a reference for creating a user library.

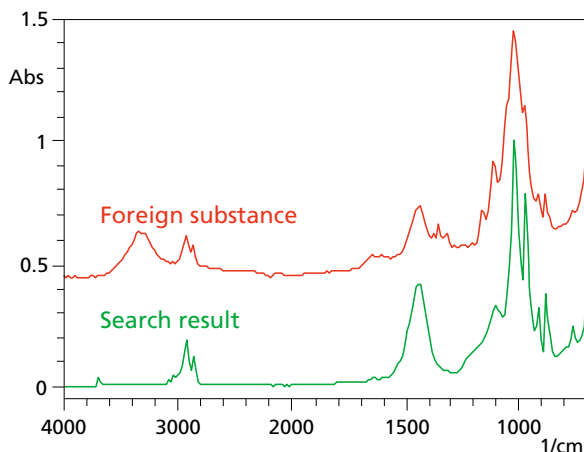
The library of foreign substances in the water supply is registered with the IR spectra for foreign substances actually collected, and commercially available water supply maintenance parts. Furthermore, it is also registered with element information obtained from analysis results using an energy dispersive X-ray fluorescence spectrometer (EDX). It is also possible to browse the details using the EDX profile database, which contains photographs of samples in addition to qualitative and quantitative information.

This section shows an example of the analysis of a foreign substance detected in the water supply using single reflection ATR measurements. The measurement conditions in this case are shown in Table 3. The IR spectral search results are shown in Fig. 1. From the search results, it is evident that the foreign substance is quite similar to the packing for a drainage fixture.

As shown here, the library for foreign substances in the water supply is a database summarizing actual foreign substances, so the search precision ratio is remarkably good. In addition to the qualitative results for the foreign material, it also has the advantage of providing information on color, form, hardness, presence/absence of metallic luster, and measurement method.

Table 3 Instrument and Analysis Conditions

Instrument:	IRAffinity-1S, MIRacle10 (Diamond)
Resolution:	4.0 cm ⁻¹
Accumulation:	40
Apodization:	Happ-Genzel
Detector:	DLATGS



Detailed Information on Search Result from an IR Spectral Database:
Packing for a drainage fixture for a 32 mm dia. pipe_gray_interior

Material: Styrene butadiene styrene (SBS),
Calcium carbonate (CaCO₃),
Magnesium silicate (TALC, 3Mg₄SiO₂H₂O)

Major Elements: Cl, Ca, Si, Mg, S, Zn, Ti

Color: Gray

Form: Resin/Ring shaped

Hardness: Soft

Metallic Luster: No

Measurement Method: ATR (diamond)

Fig. 1 Foreign Substance IR Spectrum and Search Result, and Database Details

5. Effectiveness of Advanced Searches

One disadvantage of spectral searches is the issue that unregistered substances will not produce a hit (because there will not be similar spectra). User libraries in particular only contain IR spectra registered by the user, so there are not many registered substances. Therefore, if unexpected substances are analyzed, they tend not to produce hits.

Furthermore, even if multiple similar spectra are identified, significant differences between the IR spectra are not always found. In such cases, if classification is impossible using just the IR spectra, advanced searches supplemented with information other than the IR spectrum are effective.

Normally, specifying an unknown sample is a comprehensive assessment incorporating the sample's condition including form and color, as well as other information from analysis instruments.

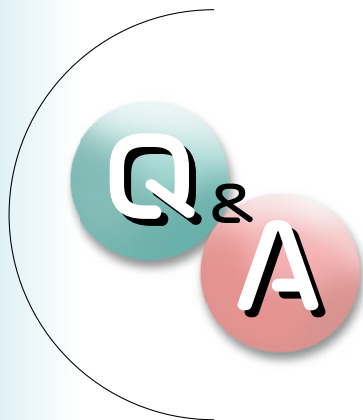
The advanced search method combines spectral searches based on IR spectra with searches of text from other information.

Registering sample information in user libraries is very effective for this sort of application as well.

6. Summary

There are many commercially available databases, more than 220,000 at present. Here, however, we have introduced the effectiveness of user libraries.

User libraries take considerable initial effort to create, but once they are created, they become important databases. It can also be effective to start by registering the samples you have at hand, and then registering the IR spectra frequently encountered in sequence as the need arises. We encourage you to create original databases to use in your daily procedures.



Question

How are the lower limits of quantitation and the detection limits determined?

Answer

The lower limit of quantitation and detection limit are the lower limit values for concentration that can be quantified and detected. Generally, a blank sample is repeatedly measured. The concentration with respect to the absorbance at ten times the resulting standard deviation is taken as the lower limit of quantitation, and the concentration with respect to the absorbance at three times the standard deviation is taken as the detection limit¹⁾. As an example, the lower limit of quantitation and detection limit were obtained from the quantitation of fatty oil via the single reflection ATR method introduced in Shimadzu Application News No. A468.

Standard samples of commercially available salad oil mixed with liquid paraffin at 0.0, 5.0, 7.5, 10.0, 12.5, and 15.0 % (w/w) concentrations were measured, and a calibration curve of the fatty oil was created. Fig. 1 shows the absorption peaks for the fatty oil. Refer to the Application News for the analysis conditions and other details. Fig. 2 shows the calibration curve created from the peak height in the vicinity of 1749 cm⁻¹. Table 1 shows the results of repeatedly measuring the 0.0 % fatty oil standard sample as a blank sample ten times, and then determining the peak height, in a similar way as for the calibration curve. The standard deviation σ from the results of the ten repeated measurements was determined to be 0.000275. The concentration corresponding to the 10 σ peak height is taken as the lower limit of quantitation, and the concentration corresponding to the 3 σ peak height is taken as the detection limit. Accordingly, from the formula for the calibration curve in Fig. 2, the lower limit of quantitation was determined to be 1.1 % and the detection limit was 0.33 %.

Lower limit of quantitation = 10 σ / Slope of the calibration curve
 = 10 * 0.000275 / 0.002486 = 1.1 %

Detection limit
 = 3 σ / Slope of the calibration curve
 = 3 * 0.000275 / 0.002486 = 0.33 %

Note that the light absorption coefficient differs with the substance being analyzed, so be aware that the calculated results for the lower limit of quantitation and detection limit will substance dependent.

Reference:

1) Michihisa Uemoto, *Bunseki* (Analysis), May 2010.

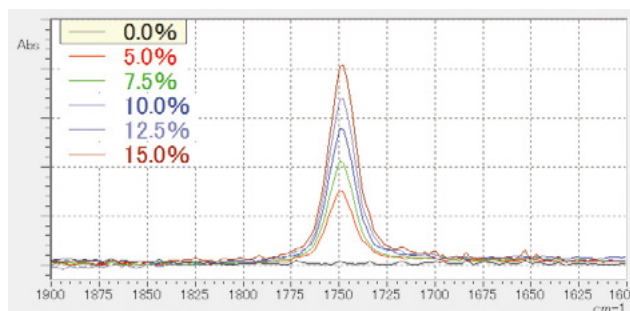


Fig. 1 Absorption Peaks for the Fatty Oil Standard Samples (Enlarged View of 1600 to 1900 cm⁻¹)

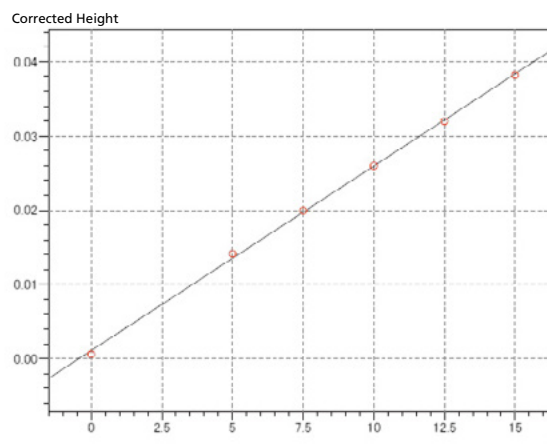


Fig. 2 Calibration Curve for Fatty Oil in Standard Samples

Table 1 Peak Height and Standard Deviation σ When a 0.0 % Concentration Standard Sample Is Repeatedly Measured Ten Times

No.	Peak Height (Absorbance)
1	-0.00015
2	-0.00008
3	0.00004
4	0.00021
5	0.00045
6	0.00003
7	0.00001
8	-0.00011
9	0.00061
10	0.00059
Standard deviation σ	0.000275



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