Technical Overview

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The Agilent Cary 630 FTIR Spectrometer for Material Identification Applications

Using Agilent MicroLab software for FTIR to perform fast, easy, and reliable workflows for the analysis of different sample-types

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Introduction

Material identification is a critical quality assurance or safety control analysis that is widely performed in many applications and industries. Confirming the identity of received starting materials helps to prevent contaminated, counterfeit, and incorrectly labeled materials from proceeding into production. The analysis ensures that the final product meets the quality and purity required for its intended purpose. It is equally important to confirm the identity of the final product to avoid errors in product packaging and labeling. Therefore, material identification is a requirement that is outlined in industry standards such as most global pharmacopeias.

Fourier transform infrared spectroscopy (FTIR) is widely used for material identification studies and is specified in most pharmacopeias for that purpose. It is a powerful technique that is quick and easy for users to operate confidently, enabling them to determine the identity of compounds.

Determining the identity of an unknown compound using a suitable analytical technique can help to investigate contamination or production problems. The analysis can also be used to identify hazardous or illegal substances such as toxins, explosives, or street drugs to inform first responders and law enforcement.

Because of the usefulness of the data, material identification workflows are routinely performed on various samples by nonexpert users. It is important, therefore, that the analysis is fast, simple, and reliable, and that the results are easy to understand, with no risk of misinterpretation.

The Agilent Cary 630 FTIR spectrometer is a flexible, high performance, and compact benchtop instrument (Figure 1). Its modular design allows laboratories to configure the analytical system for a variety of sample-types and FTIR applications. The range of interchangeable sampling modules for the Cary 630 FTIR include transmission, DialPath, TumblIR, diamond attenuated total reflectance (ATR), germanium ATR, zinc selenide ATR, zinc selenide multibounce ATR, specular reflectance, and diffuse reflectance.

The Cary 630 FTIR spectrometer is controlled using Agilent MicroLab software, which uses a pictorial interface to guide users through the steps of the analysis from sample introduction to reporting (Figure 2). The software automatically detects which sampling accessory is installed, applies the required settings, and loads instructive images that are specific to the sampling accessory. The results are color-coded to help users interpret the data and to take any appropriate action.

Figure 1. The Agilent Cary 630 FTIR with diamond ATR sampling module is used to establish the identity of different powders.

This technical overview outlines different approaches to material identification by FTIR. Application examples show how the Cary 630 FTIR with MicroLab software can be used to perform fast, easy, and reliable material identification workflows of different sample-types.

Figure 2. The intuitive Agilent MicroLab software workflow makes finding answers with the Agilent Cary 630 FTIR spectrometer simple. The picture-driven software also reduces training needs and minimizes the risk of user-based errors.

Ways to identify an unknown substance or confirm the identity of a sample

Library search

Each substance has a unique IR spectrum (expect for some compounds such as optical isomers and long chain alkane homologues) that can be considered to be a fingerprint of the substance. Comparing the IR spectrum of an unknown sample with a library of spectra of known compounds allows the unknown sample to be identified (when the unknown sample is part of the spectral library), see Figure 3. Identifying an unknown substance through a "fingerprint" match with a compound-spectrum in a spectral library is called positive identification.

A spectral library can contain tens, hundreds, or sometimes even thousands of spectra. Manual comparison against all these spectra to find the best match is often not feasible. Therefore, search algorithms have been developed that automatically identify the best match. The confirmation of the identity of a material does not necessarily require a large library with a high number of spectra. Smaller libraries that contain only spectra of potential materials are often sufficient for the intended application. Irrespective of the size of the library, library search algorithms simplify the confirmation of materials with its "matched" identity.

A hit quality index (HQI) is automatically calculated for each library item. The HQI value indicates how well the measured spectrum and the library spectrum match. The HQI is often used as pass/fail criteria in material identification and confirmation workflows.

Library search with the Agilent MicroLab software

The MicroLab software, which is provided with the Cary 630 FTIR spectrometer and the Agilent mobile and handheld FTIR systems, uses a method-based approach. Once the method is set up, as described in the next section, the software turns the FTIR system into a turnkey solution that enables quick decision-making.

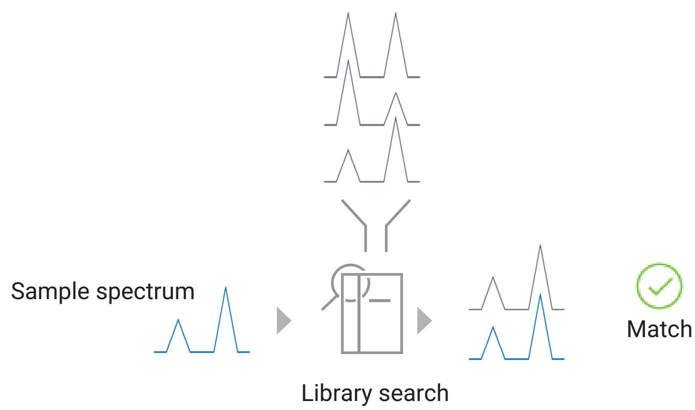


Figure 3. Comparing the FTIR spectrum of an unknown sample with spectra of known compounds allows identification of the unknown. This workflow is called "Library Search" and can be automatically performed using the Agilent MicroLab software.

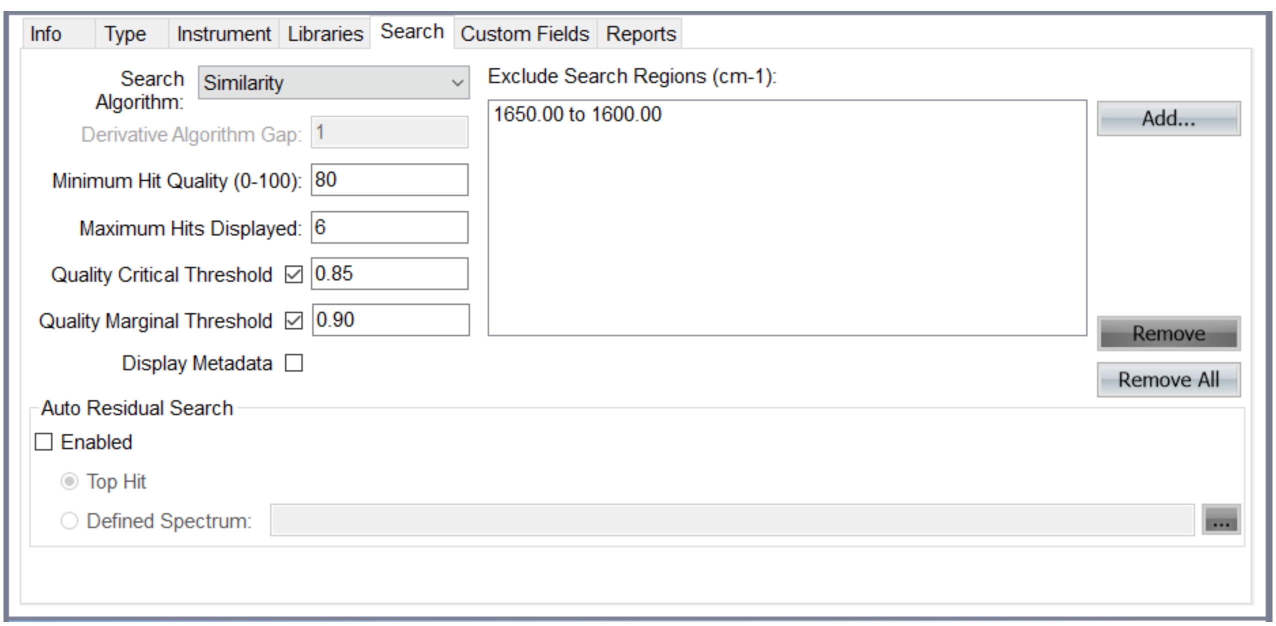


Figure 4. In the Agilent MicroLab software, the library search method and search parameters can be tailored to the specific identification application. The results-display settings are also customizable, so that the results are easy to interpret and report.

Method setup

During method setup, the library search method can be tailored to the identification application:

- Multiple libraries can be searched simultaneously, e.g., one library with reference standards to identify the sample and another library with out-of-specification samples to identify re-occurring problems.
- A broad selection of search algorithms is available (Euclidean, absolute value, derivative absolute value, least square, derivative least square, correlation, derivative correlation, similarity, derivative similarity, and extended correlation).
- Spectral ranges can be excluded from the analysis.
- Setting a minimal HQI and maximum number of hits ensures that only relevant library hits are presented.
- Color-coding of the results based on the HQI can be used to define confidence levels, helping users to interpret the results and reducing oversights that may lead to errors.
- Additional compound-specific information can be presented (e.g., hazard information).

Performing an analysis

MicroLab uses pictures to navigate the user through each step of the analysis including sampling (e.g, how to use the ATR press for solid samples) and cleaning procedures (Figure 5). Clicking the **Start** button on the home screen of the software starts the analysis.

After data acquisition, the software automatically performs a library search and provides the user with a list of the best library matches in an easy-to-understand result display format, as shown in Figure 6.

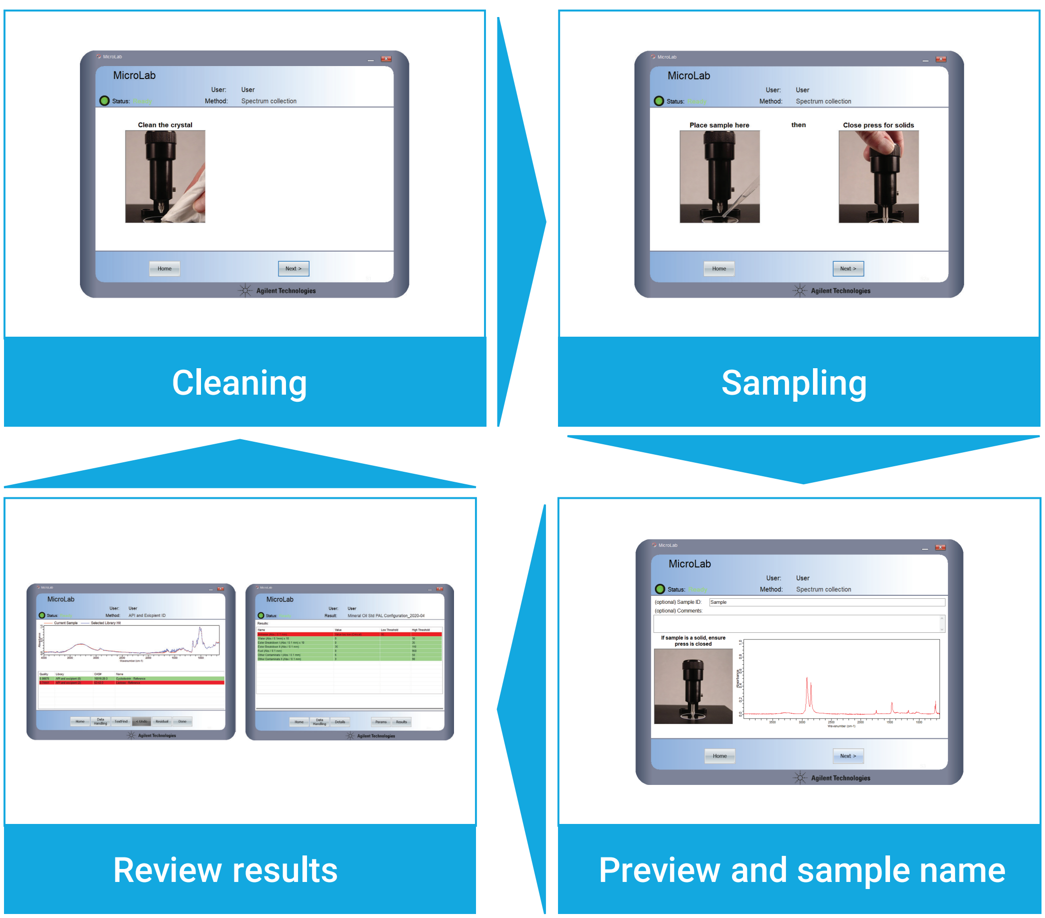


Figure 5. Agilent MicroLab software picture guidance interface simplifies performing an analysis using the Agilent Cary 630 FTIR, reducing training needs. The software replicates what the operator sees when using the instrument. After data acquisition, the results are directly displayed in an easy-to-understand format as shown in Figures 6 and 10. After the user has checked the results, the software starts the next analysis, ensuring a continuous analytical workflow.

Figure 6. In the Agilent MicroLab software, the result-screens of library search methods are easy to understand, enabling nonexpert users to review and interpret the results. Left: Library hit data is displayed as a list only or as a list plus spectra. Right: The optional display of chemical metadata including hazard information and first response information is also available

Libraries and library management

The quality of a library search strongly depends on the quality of the library. Agilent provides a wide selection of ready-to-use, application-specific libraries that can be used with the MicroLab software.

Alternatively, spectral libraries can be easily created, maintained, and managed in the MicroLab software (Figure 7). A new library can be created in a few seconds. Spectra can be added to the library, either at the time of creation or at any other time, directly from the results screen.

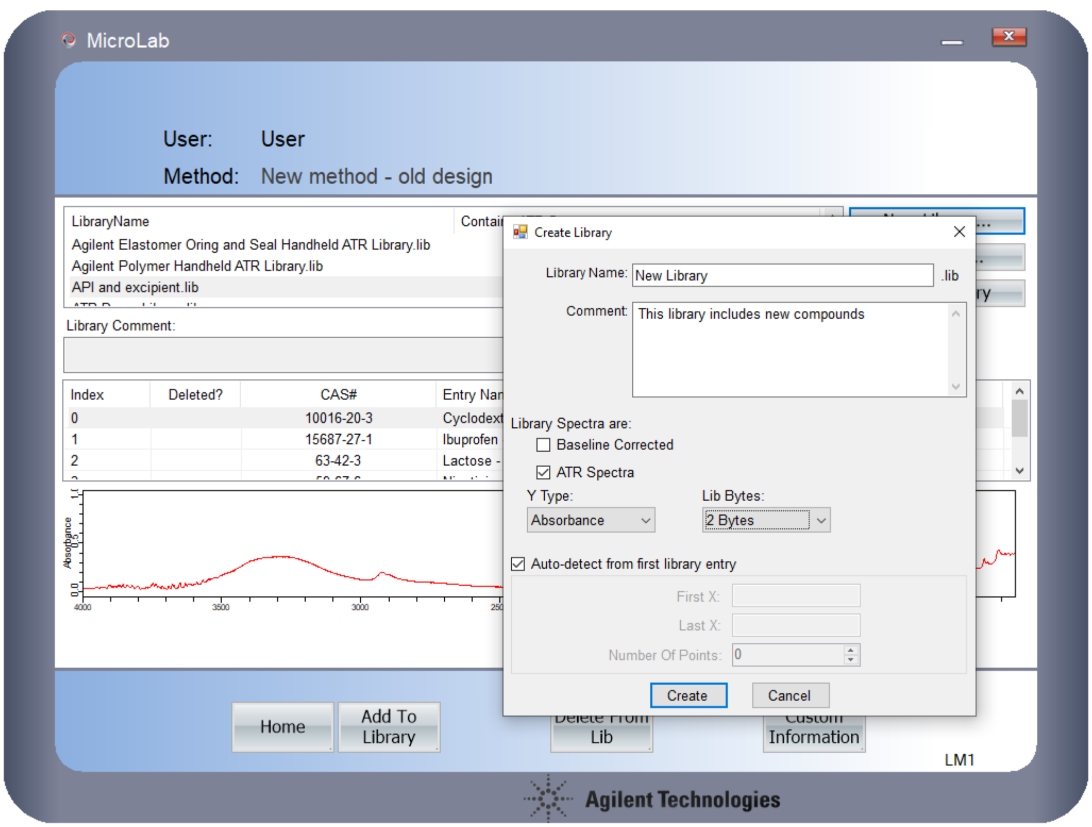


Figure 7. Library management in the Agilent MicroLab software is quick and easy. New libraries can be created in a few seconds and the library content can be updated as required.

Application examples

Application example 1: Material identity confirmation of pharmaceuticals

IR spectra of three commonly used active pharmaceutical ingredients (APIs), salicylic acid, ibuprofen, nicotinic acid, and an excipient (α-cyclodextrin) reference material were collected using a Cary 630 FTIR with diamond ATR sampling module. A spectral library was created in the MicroLab software and the collected reference spectra were added to the library. This spectral library was used in a library search method using the parameters given in Figure 8.

The material identity confirmation method was then used to analyze different batches of salicylic acid, nicotinic acid, ibuprofen, and α-cyclodextrin. The MicroLab software correctly confirmed the identity of all tested materials. The HQIs were 0.99238 (salicylic acid), 0.99775 (nicotinic acid), 0.98641 (ibuprofen), and 0.98675 (α-cyclodextrin), respectively, where 1 is the highest theoretical value using the similarity search algorithm (Figure 9).

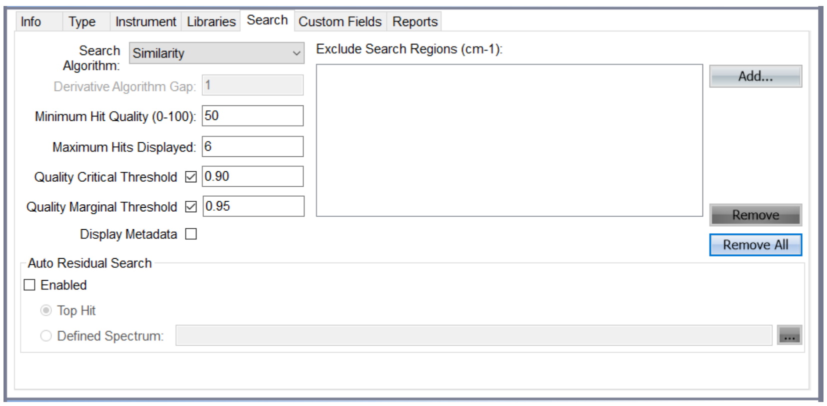


Figure 8. Library search parameters used to confirm the identity of pharmaceutical APIs. The Quality Critical Threshold and the Quality Marginal Threshold settings define the color-coding in the results-display.

Figure 9. The Agilent MicroLab software applies color-coding to the library search results. Color-coding simplifies the interpretation of the results and reduces oversights that may lead to errors. The screenshot shows the results for α-cyclodextrin in green, indicating a good match with the library spectrum.

Example application 2: Identification of polymer samples

The Cary 630 FTIR with diamond ATR sampling module was used to analyze five different plastic materials, including polypropylene, polycarbonate, chlorinated polyethylene, poly(ethylene terephthalate), and poly(vinyl chloride).

The Agilent FTIR spectral library *ATR Polymers and Polymer Additives* (part number G8045AA, option 106) was selected in the Agilent MicroLab software. The spectral library contains 7,974 spectra of selected polymers, plastics, polymer additives, plasticizers, and packing materials.

Using the similarity algorithm to search the spectral library, all five polymer samples were correctly identified, as indicated by the HQI results. The HQI values were 0.98315 (polypropylene), 0.97298 (polycarbonate), 0.97212 (chlorinated polyethylene), 0.96238 (poly(ethylene terephthalate)), and 0.98360 (poly(vinyl chloride)), with 1 being the highest theoretical value when using the similarity algorithm (Figure 10).

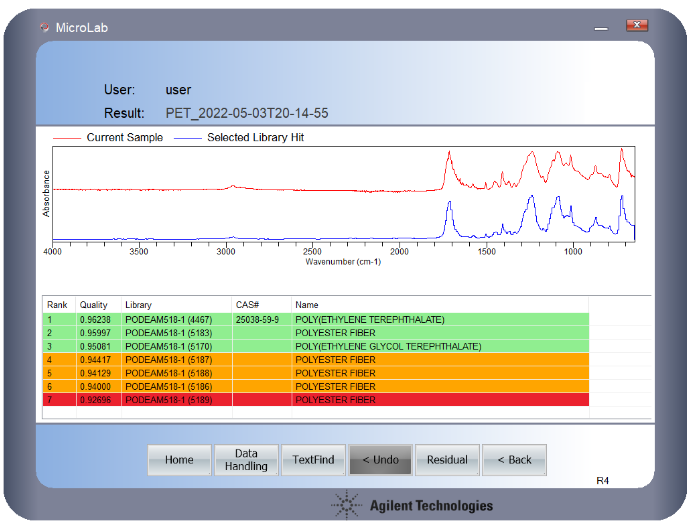


Figure 10. Color-coded matching results obtained using the Agilent Cary 630 FTIR with diamond ATR sampling module are displayed quickly and clearly. A routine, qualitative, material identification method can automatically identify the type of polymer with a high confidence level.

Manual comparison of spectra

The identity of a known material can also be confirmed by a manual comparison of the IR spectrum. The spectrum of the sample is either visually compared to a spectrum of a reference material or the observed peaks are labeled and compared to literature values.

Typically, most spectral information is found in the fingerprint region (approximately 400 to 1,500 cm^{-1}) of an IR spectrum. The MicroLab software allows the wavenumber scale of this fingerprint region to be expanded while compressing the higher energy region of the spectrum (Figure 11). Expanding the spectrum enables a deeper and easier investigation of the more relevant fingerprint region.

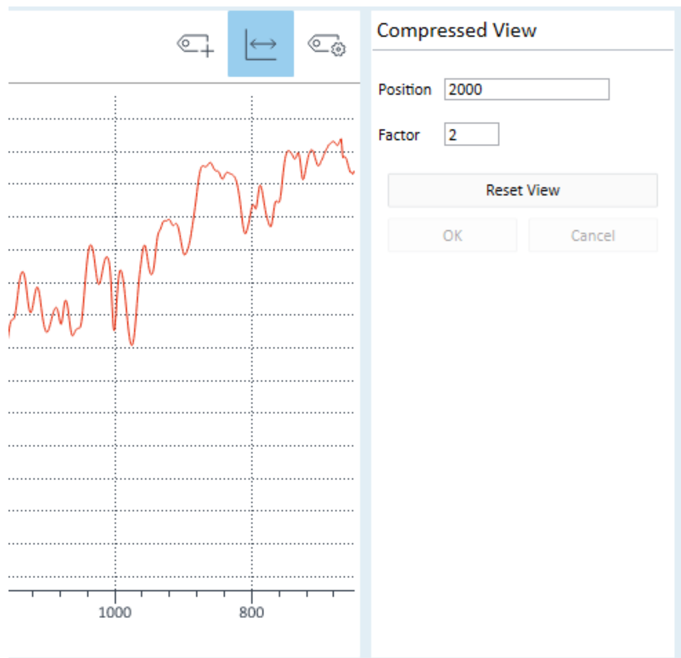


Figure 11. The IR spectrum of a substance can provide valuable insights into the composition of a material, such as the presence of functional groups. Often the most relevant bands are found in the fingerprint region (approximately 400 to 1,500 cm^{-1}) of the IR spectrum. In the Agilent MicroLab software, a wavenumber scaling factor can be applied, enabling a more detailed analysis of the spectral range of interest.

Application example 3: Material identity confirmation of an API by manual spectral comparison

An IR spectrum of a potassium bromide (KBr) pellet containing rifampin (an antibiotic API), was collected using a Cary 630 FTIR with transmission sampling module. Little spectral information was observed in the range between 4,000 and 2,000 cm^{-1}, while most bands were found in the region between 2,000 and 600 cm^{-1}. On the graphic result screen in the MicroLab software, the more important fingerprint region was expanded (by a factor of two) while the spectrum at higher wavenumbers was compressed (Figure 12).

Expanding the spectrum allowed for an easy comparison of the sample-spectrum with a reference spectrum in the Atlas of infrared spectra of drugs issued by the Chinese Pharmacopeia Commission. The relevant bands were labeled, and the software automatically generated a PDF report that included the spectrum and a table of all labeled peaks (Figure 13).

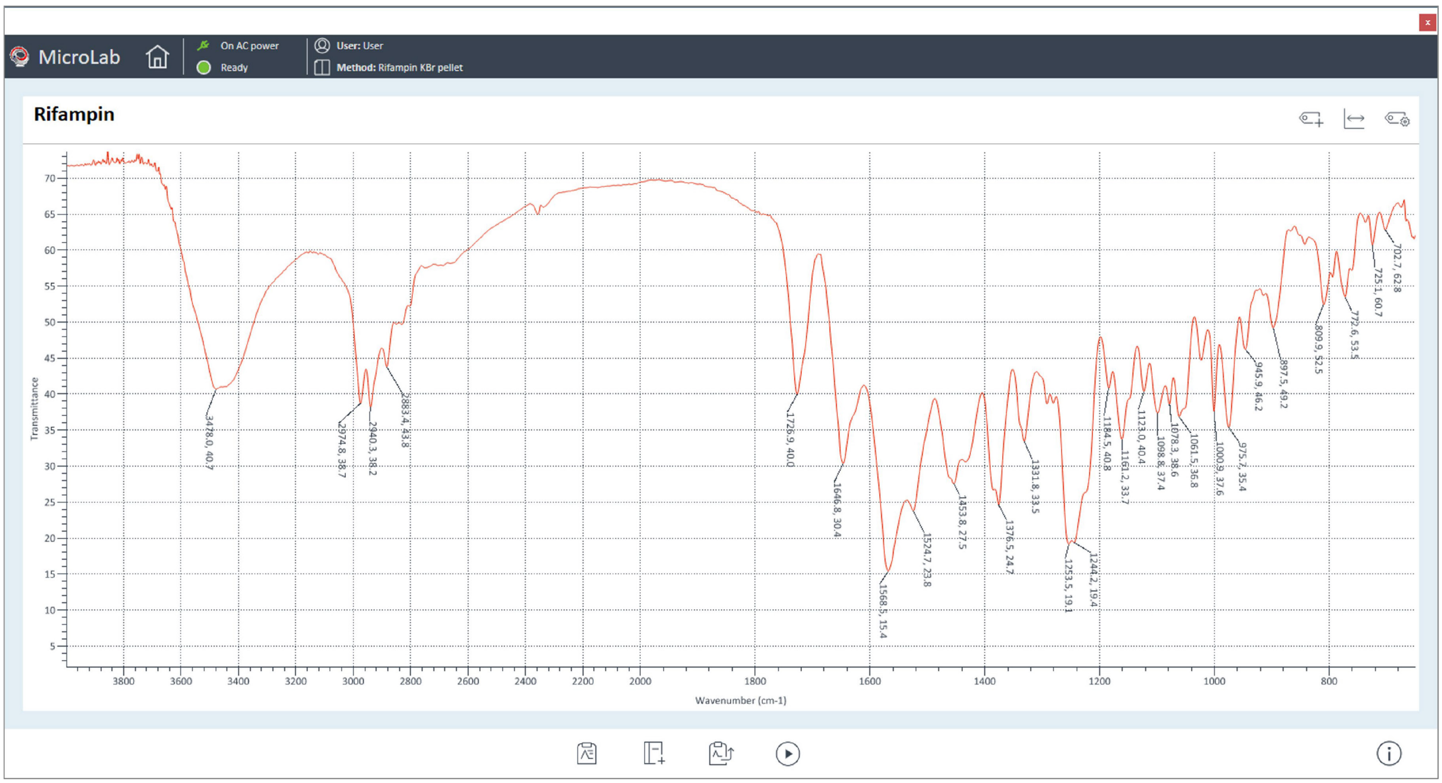
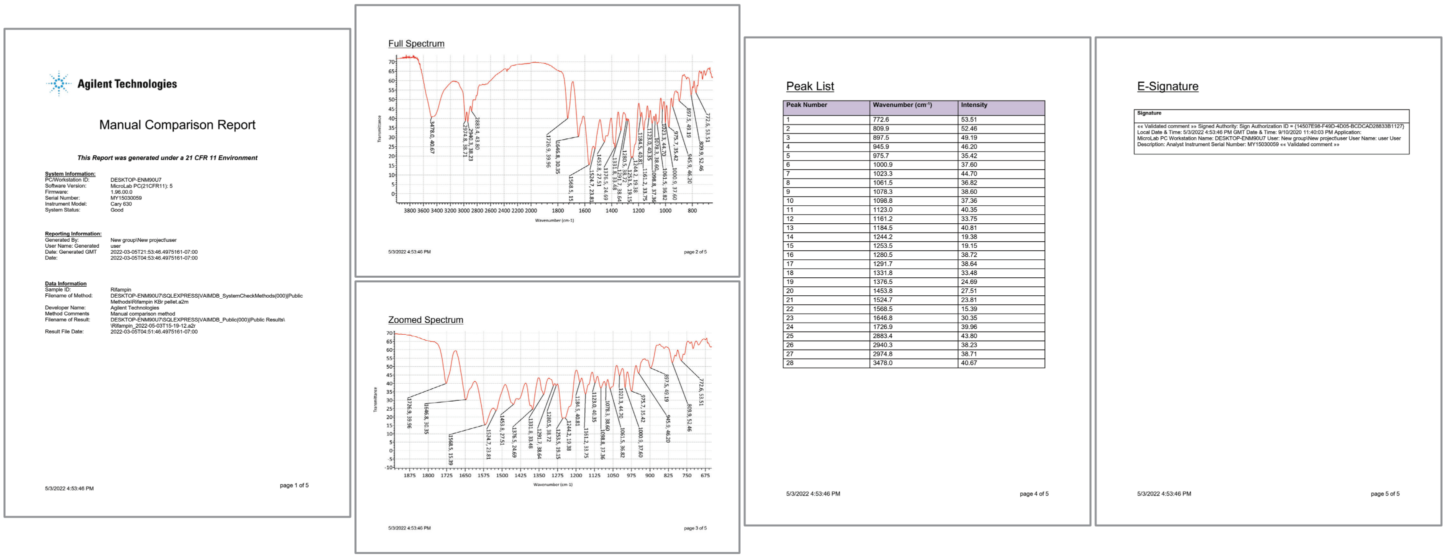


Figure 12. IR spectrum of rifampin. To help the review of the spectrum, the fingerprint region was expanded by a factor of two compared to the higher wavenumber region, and the relevant bands were labeled.



Peak Number	Wavenumber (cm^{-1})	Intensity
1	772.6	53.51
2	809.9	52.46
3	897.5	49.19
4	945.9	46.20
5	975.7	35.42
6	1000.9	37.60
7	1023.3	44.70
8	1061.5	36.82
9	1078.3	38.60
10	1098.8	37.36
11	1123.0	40.35
12	1161.2	33.75
13	1184.5	40.81
14	1244.2	19.38
15	1253.5	19.15
16	1280.5	38.72
17	1291.7	38.64
18	1331.8	33.48
19	1376.5	24.69
20	1453.8	27.51
21	1524.7	23.81
22	1568.5	15.39
23	1646.8	30.35
24	1726.9	39.96
25	2883.4	43.80
26	2940.3	38.23
27	2974.8	38.71
28	3478.0	40.67

Figure 13. A PDF report can be generated directly from the results window of the Agilent MicroLab software. The report is fully customizable to include all relevant information, e.g., sample name, operator identity, and analysis details, including the zoomed and full spectrum, and peak list.

Conclusion

Material identification by FTIR is an important analysis in many industries. The overview showed the flexibility of the Agilent Cary 630 FTIR for material identification of diverse sample-types, using a sampling module and method tailored to the application. Any method development was simplified using the Agilent MicroLab software that provides step-by-step guidance through the workflow. Application examples were provided showing how the software facilitates material identification through library searches or manual comparison of spectra.

The intuitive, picture-guided MicroLab software is quick and easy to learn, reducing training needs, operator-errors, and reporting times—all factors that increase the productivity of quality control testing of materials.

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