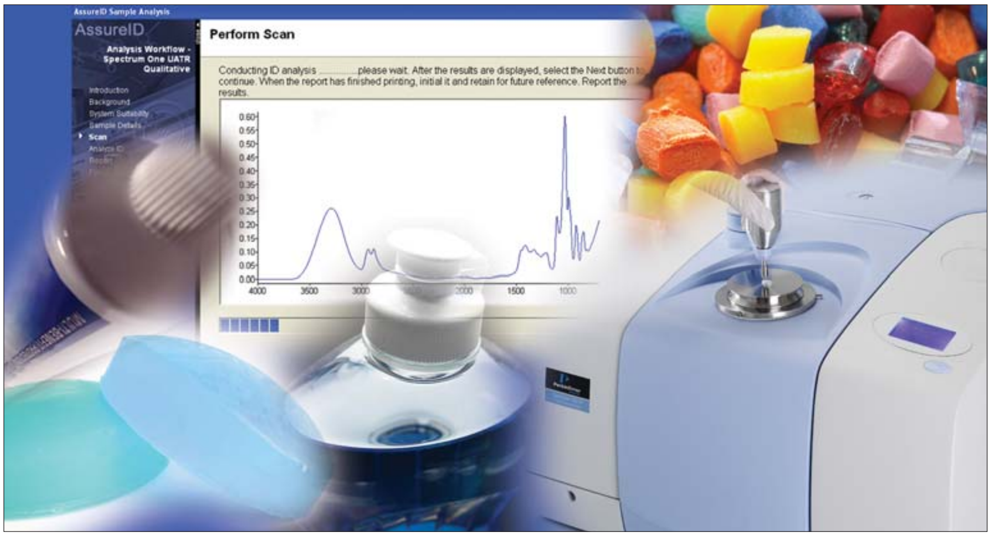
CASE STUDY

INFRARED SPECTROSCOPY

Infrared Fingerprinting Assures Quality, Saves Time



Thousands of shipments of raw materials arrive every day at manufacturing plants around the world operated by a major consumer goods producer. The company assures compliance with its strict internal quality standards by generating a unique fingerprint of incoming shipments using Fourier transform infrared spectrometry (FT-IR). Any change in the incoming material from what has previously been approved is immediately detected and the material is not accepted unless it can be proven that it will not have a negative impact on the finished product. The use of automated instruments and software makes this unique level of inspection cost-effective by allowing users without scientific training to generate infrared spectra from a sample and validate them against reference spectra in about five minutes. The ability to quickly provide positive identification of incoming raw materials helps the company maintain the highest possible quality standards while saving time that can be applied to other quality control procedures.

For decades, the consumer goods producer has led the way in the personal care industry by carefully testing incoming raw materials. Testing raw materials doesn't eliminate the need for testing the finished product but it does help avoid waste in the manufacturing process and provides an extra level of quality assurance. When the company first started this program, it used conventional analytical methods such as liquid chromatography to analyze incoming shipments. The length of time required to perform the analysis made it impossible to validate every incoming shipment and a very small potential for error existed since, in some cases, different materials can generate indistinguishable chromatographs.

Infrared fingerprinting provides positive identification

For these reasons, the company became one of the first to begin generating infrared spectra of incoming materials using dispersive infrared spectrometers. Just like human fingerprints uniquely identify their owner, infrared spectroscopy provides a spectral fingerprint that uniquely identifies a chemical compound. Fingerprinting is superior to other analytical methods because no two compounds have the same infrared spectrum. This is a totally unique technology and the company is continually finding new uses for it.

In the mid 1980s, as the company was planning the switch from dispersive IR to FT-IR, it evaluated four different vendors and selected a PerkinElmer FT-IR instrument. It is still using that first instrument, purchased over 20 years ago. Since then, the group made the decision to standardize on PerkinElmer FT-IR instruments throughout its global manufacturing operations and has established a global contract with PerkinElmer for FT-IR instruments and software. The advantage of this approach is that the different plants, using the same equipment, can share knowledge, files, libraries and problem-solving capabilities. The company has continued to purchase PerkinElmer FT-IR instruments and now uses them as its global standard at over 20 facilities throughout the world based on their accuracy, dependability, ease-of-use and the strong partnership that has been developed over the years.

Lengthy manual procedures required in the past

In the early days, generating infrared fingerprints was a time-intensive process that required the skill of a scientist to ensure the integrity of the results. Scientists prepared samples for analysis by mixing a small amount of the sample with a large amount of potassium bromide, which has no IR spectrum. Then the mixture was placed in a stainless steel press and three tons of pressure was applied until the powder is liquefied. When the pressure was released, the material turned back to a uniform mixture of two powders. Liquid samples, on the other hand, were placed between two potassium bromide disks. The complexity of the instruments meant that they had to be used by scientists who were trained in their operation. A minimum of three to four hours was typically required to generate infrared spectra from the samples and compare them to spectra generated by good material in the past. With some plants receiving 30 to 40 shipments per day, a large staff of scientists was required to keep up with the incoming material workload. The truck drivers waited while these tests were performed.

Based on early successes, the company established a standard in which every shipment of raw materials is fingerprinted and compared to a standard to ensure the quality of the finished products. It also made the decision to establish its own internal quality standards for purchasing, manufacturing, packaging and other areas and established an internal audit team to ensure that each operation complies with these guidelines. The company experimented with different equipment and methods in an effort to increase the speed of their fingerprinting process without sacrificing accuracy.

Automating the process of generating infrared spectra

The company used PerkinElmer® Spectrum™ Procedures software to develop operating procedures that automate much of the process of performing FT-IR analysis. It set up entire processes for FT-IR analysis ranging from preliminary system checks, data collection, analysis and report generation. These processes took advantage of the fact that Spectrum Procedures software enables the use of templates to quickly build useful procedures without programming and also provides a flexible and open architecture for more complex procedures.

The user starts up the program and enters their name and password. The program then prompts the user for the material that is being fingerprinted and the lot number. The program then checks the diamond sampling surface to make sure it is clean. Then it prompts the user to place the sample on the diamond. When the user says the sample is ready, the software then operates the instrument to collect the spectrum and compares it to a reference spectrum for that material. The reference spectra is determined by examining six different lots over a period of time in order to capture process variations. The PerkinElmer compare function calculates a correlation coefficient between the sample and the reference spectra. If the sample passes the test, then the shipment is accepted. The report prints out for the operator and the results of the test are automatically archived. The entire process takes only five minutes, not even enough time for the truck driver to finish a cup of coffee.

Further analysis performed if spectra do not match

If the sample correlation is not high enough to pass the test, then a hold is placed on the material and additional tests are performed by scientists in the laboratory to determine what the problem is. They will check the instrument used to perform the initial test and also perform other tests to determine what is different about this material. The final decision of whether or not the material is acceptable is made by the person responsible for the formula.

Another major advancement has been the adoption of the diamond ATR accessory that reduces the time required for and simplifies sample preparation. This device handles samples such as abrasives, resins, corrosive liquids, powders and other intractable samples. For hard, intractable solids or powders, a pressure applicator forces the sample against the diamond for optimal contact. No further sample handling is required. The analysis time, including sample positioning and spectral collection, is usually less than 30 seconds. For liquids, a drop of liquid is placed on the diamond and held in place by surface tension. For volatile samples, a volatiles cover is provided. Films are also easily cast on the diamond for analysis of solvent extracts such as solvent washes, LSD analysis, etc.

Automated methods save time

The global approach saves time in method development and has helped reduce the time required to analyze incoming shipments from 3 hours to 5 minutes. With 25 of the company's plants now using these methods to inspect 3 to 40 shipments per day, it's possible to estimate that the company is saving something on the order of 1500 hours per day. The other plants are operating on a smaller scale in areas where the cost of labor is so cheap that they currently cannot justify these advanced methods. The time savings are equivalent to more than 150 full time positions. These numbers are a rough approximation but the basic idea is clear. The company is able to maintain the highest possible quality standards while freeing up a considerable amount of time for other quality improvement projects.

The consumer products manufacturer continues to develop methods for providing even higher levels of quality assurance and further time savings. It is working on procedures to fingerprint finished materials which will provide an extra margin of safety while eliminating the need for some other tests. It is planning to implement PerkinElmer's new Spectrum AssureID™ materials checking system that increases the level of automation that can be integrated into the infrared fingerprinting process.

ANALYZER WORKFLOW SCREEN CAPTURES

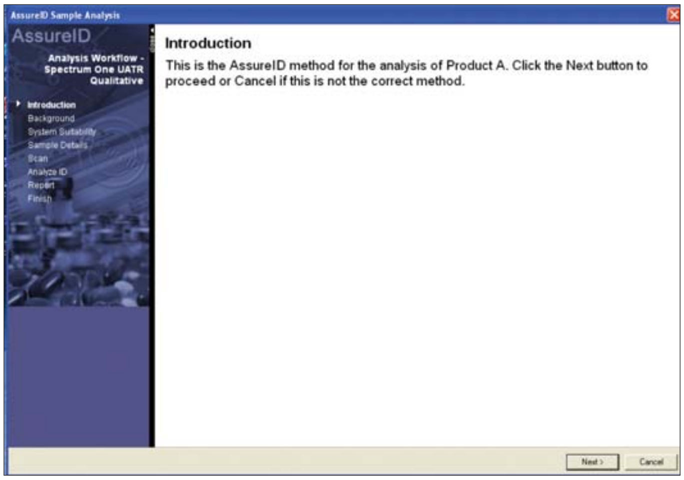


Figure 1. Analyzer Mode Page 1 - Introduction

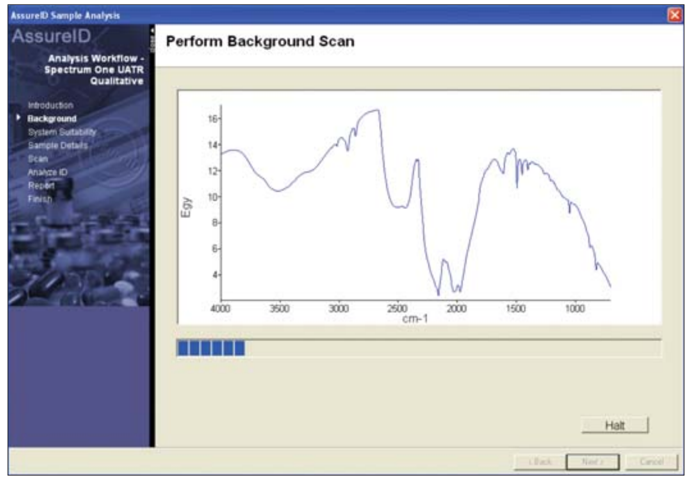


Figure 2. Analyzer Screen #2 – Scanning the background

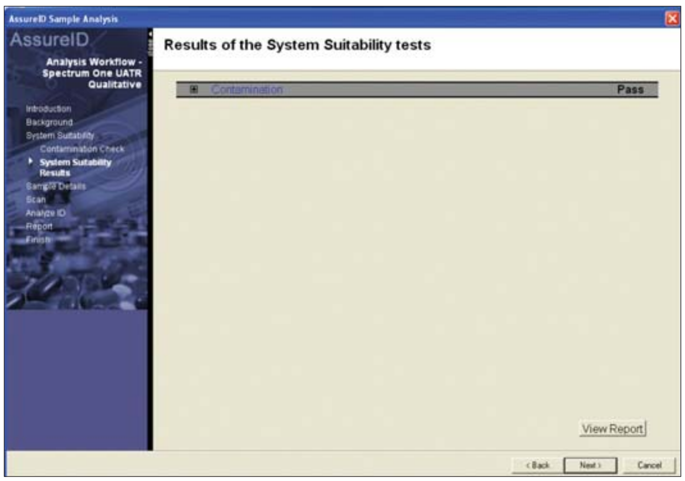


Figure 3. Analyzer Screen #3 – System Suitability Testing

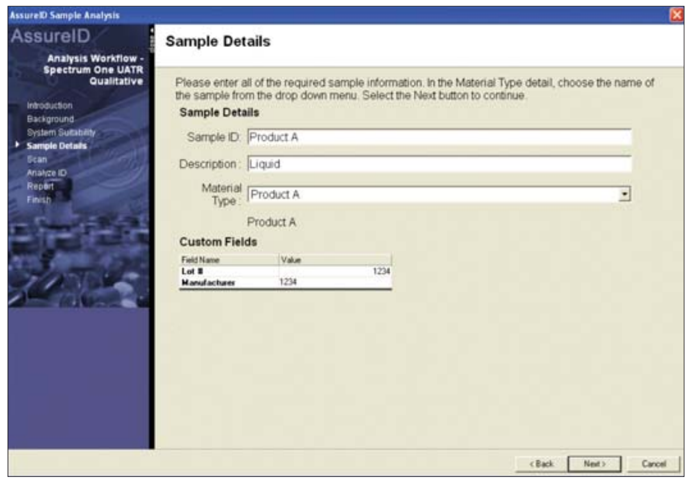


Figure 4. Analyzer Screen #4 – Sample Details

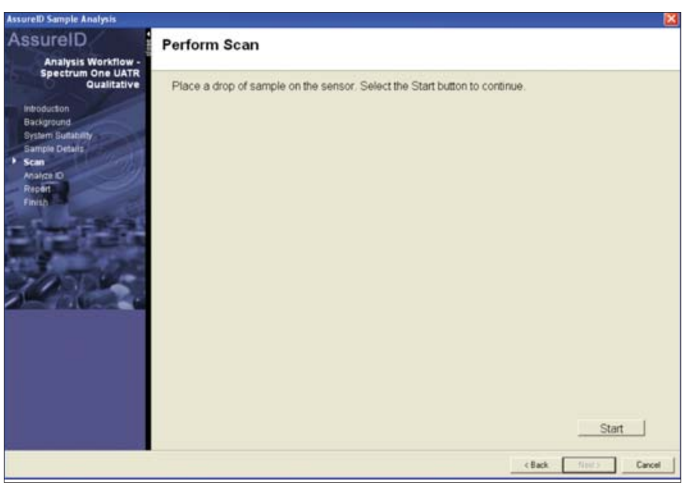


Figure 5. Analyzer Screen #5 – Sample Preparation

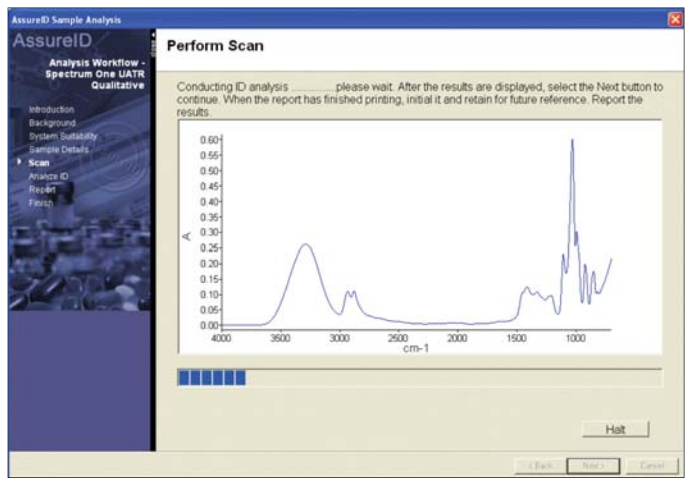


Figure 6. Analyzer Screen #6 – Scanning the sample

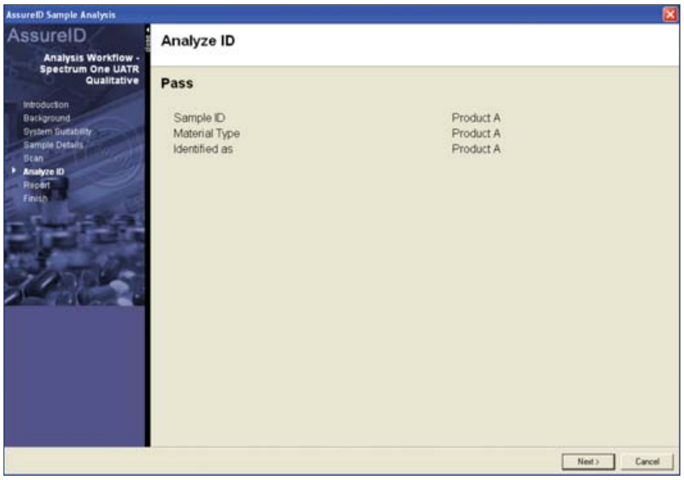


Figure 7. Analyzer Screen #7 – Raw Material Identification Results

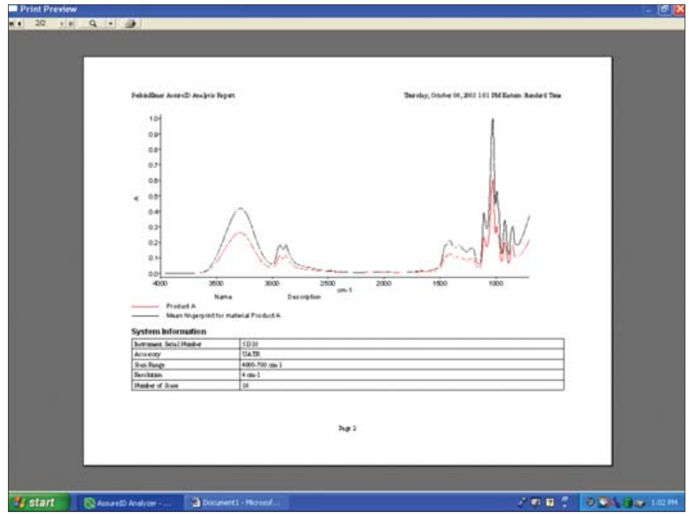


Figure 8. Analyzer Screen #8 – Results printout

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