Powerful New Identification Tools with OMNIC Specta Software

Key Words
FT-IR, Infrared, Mixture Identification, Multi-component Analysis, Raman, Spectral Searching

Introduction

Identification of materials is one of the most common applications of FT-IR. A simple (pure) material can be processed from sample preparation to identification in seconds using ATR sampling and macros to structure the SOP. However, many analytical problems are more complex, with the need to identify multiple components or contaminants within a single sample.

Under normal circumstances, mixture analysis requires either trained insights into the spectrum or a multi-step deconstruction procedure. The latter begins with a spectral search done against the appropriate library of spectra. The spectrum for the best-match compound is moved to the workspace, and a subtraction is completed using a variable scaling factor k (Original – k*Match). The process can then be repeated, with the residual spectrum being searched against the same or a different spectral library, and another best-match chosen. Depending upon signal-to-noise and the limitations of the processing algorithms, this operation may be able to extract three components from an ideal mixture.

While this procedure can function very well, it has several drawbacks. First, the user must select the appropriate match from the first search. In some cases, this may not be the top result, which requires further insight beyond the simple search. Second, the magnitude of the subtraction scaling factor requires a judgment call by the user, to determine when the component has been adequately removed. The results of the second search can be distorted by poor choice of k or the presence of artifacts, and the distortions are exacerbated with further iterations. It is difficult for two users to obtain exactly the same results at the end, especially as regards the k-factor.

Finally, there are often small peak shifts and line width changes caused by mixing. For instance, water added to acetone shifts the carbonyl peak and broadens it, due to hydrogen bonding. These intermolecular interactions can cause large derivative-shaped residuals in the subtracted spectrum, especially for strongly absorbing peaks. These residuals must either be flat-lined or ignored in successive searches.

Thermo Scientific™ OMNIC™ Specta™ software offers a breakthrough in mixture analysis. Using a powerful algorithm, OMNIC Specta makes no assumptions about the top hits returned from a search. Each of the best-matches is combined with the entire database in a second iteration, with the weightings of the spectra returned. The top pair matches may then be combined with the entire database for a third pass, and then even a fourth iteration can be completed.

Thermo SCIENTIFIC

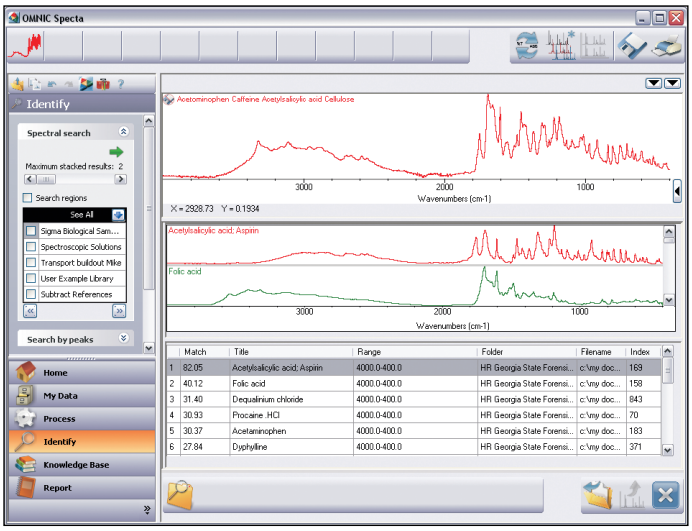

Figure 1: Normal results from search on a pharmaceutical mixture. The top hit is correct, but the overall appearance is not satisfactory. Note one of the other components is listed.

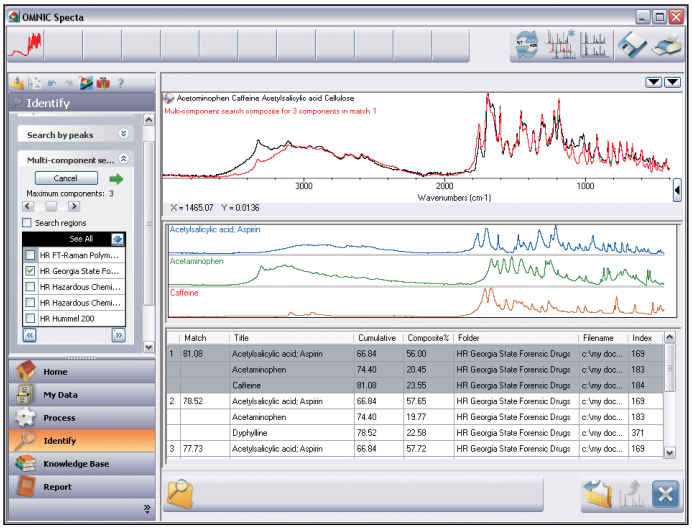

Figure 2: Multi-component search results from the same mixture as in Figure 1. All of the ingredients are correctly identified.

This multi-component searching algorithm avoids the drawbacks of the search-and-subtract method. Since multiple best-match spectra are used at each step, the final result represents a much better sampling of the possible compositions than the best-match only. Accidental top hits are quickly reduced in importance as combination results provide better agreement. Automation of the procedure means there is no k-factor choice to be made, resulting in consistent results from all operators.

The speed of the processing depends directly upon the size of the spectral database. With a reasonable library size, OMNIC Specta provides results in a vastly shorter time than the manual process. This, combined with the consistent results the algorithm provides, immediately leads to greater confidence in the results and higher productivity in the laboratory.

Pharmaceutical Mixtures

Many pharmaceutical dosage forms consist of multiple components, such as active ingredients and binders. For testing of OMNIC Specta with samples of this kind, the infrared spectrum for a mixture of acetaminophen, acetyl salicylic acid and caffeine was collected on a Thermo Scientific™ Nicolet™ iS™10 FT-IR spectrometer* using a diamond ATR accessory. The spectrum was processed in OMNIC Specta using our Advanced ATR Correction Algorithm.[1] The Thermo Scientific Georgia State Forensics Library was used as the database for the search. The results of a simple search are shown in Figure 1. Obviously, this is a good start. The top component is present in the mixture, and one match further down the list is also, but this information is not obvious except to the trained user. The multi-step procedure detailed above would normally be required for further analysis.

The search was repeated using the same library and the multi-component search algorithm of OMNIC Specta. The result is shown in Figure 2. The algorithm correctly identified all three ingredients. Considering that the same result would be obtained regardless of who performed the analysis, OMNIC Specta is providing unparalleled consistency.

Scanning through the next few trios of results reveals that the top two components remain the same in all cases, while the third component cycles through a few compounds similar to caffeine. Recalling the description of the algorithm given above, this shows the algorithm is stable even though it tries multiple combinations of library spectra.

The weighting factors represent the percentages of each component spectrum present in the composite spectrum. Small variation in these factors across the reported matches lends credence to the procedure, as it shows the search is trying many different combinations throughout the process, not just freezing one component after an initial search. The quantitative use of these factors is limited, since the search algorithm assumes all of the materials have the same absorptivity (the library spectra are normalized to 1). The procedure is essentially qualitative in nature, not quantitative.

The ability for an analytical lab to obtain consistent results through a fixed algorithm, rather than needing to train each operator to use a variable factor is a powerful aspect of OMNIC Specta. With minimal experience, anyone will obtain the same results.

Plastics Analysis:
Flame Retardant in a Computer Monitor Chassis

A critical question in the processing of polymers revolves around the presence of low concentration additives or contaminants. As an example, consumer plastics often contain flame retardant materials in low concentrations. Many of these retardants are tightly regulated through WEEE/RoHS legislation.[2] For testing, a small piece of plastic from the body of a computer monitor was obtained using a microplane. This was placed onto the diamond accessory in a Nicolet iS10 spectrometer*. The resultant spectrum was loaded into OMNIC Specta, and ATR and baseline corrected.

An initial search using the Hummel Polymers Library plus the Polymers and Additives Library, shown in Figure 3, yielded an ABS/Polycarbonate copolymer as a result. This is the bulk material, and the match value is very high (>90), which could lead to a false conclusion. However, visual inspection of the search results shows there are many small peaks between 1000 and 1400 cm^{-1} not accounted for.

The result from the multi-component search (with 2 components) is shown in Figure 4. The top composite hit lists the bulk polymer and the additive Tetrabromobisphenol (TBBP), a flame retardant material. Again, the base plastic spectrum matches the major peaks, and now the TBBP spectrum accounts for the rest. The visual comparison is stunning – the resulting composite spectrum is almost perfect.

The next few matches are very instructive. First, the base polymer spectra (the first in each pair) are similar – they are from slightly different formulations. The second match in every case is TBBP. This reinforces the idea that OMNIC Specta is sampling every possible combination, and is returning consistent information.

Deformulation:
Thermo Gravimetric Analysis – Infrared (TGA-IR)

Samples in a TGA are heated while suspended on a microbalance. The TGA records weight as a function of temperature – the decrease in weight is quantitatively tied to the driving off of materials from the sample. Addition of an infrared to the TGA provides qualitative information about what gases are driven off. Many times in TGA-IR, multiple gases are emerging simultaneously; normally, the sequential search-and-subtract procedure noted in the introduction is required to analyze the data. However, since gas phase spectra are almost perfectly additive, the multi-component analysis tool in OMNIC Specta can provide spectacular results.

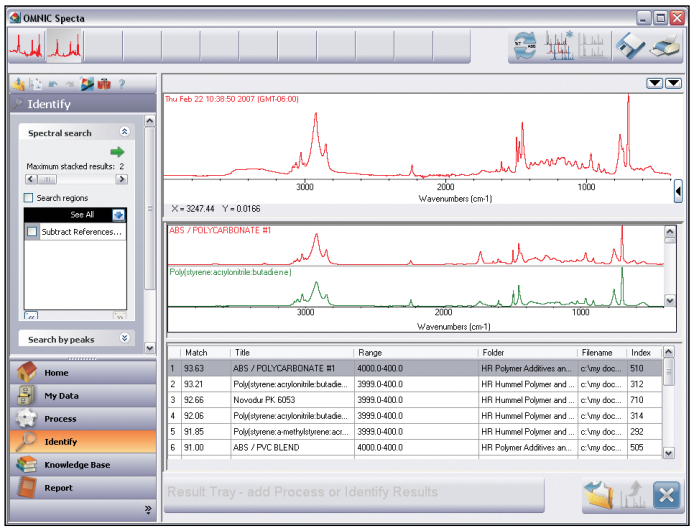

Figure 3: Normal search result for sample from monitor. All of the top results are essentially the same – the main polymer – but there are many small peaks not accounted for.

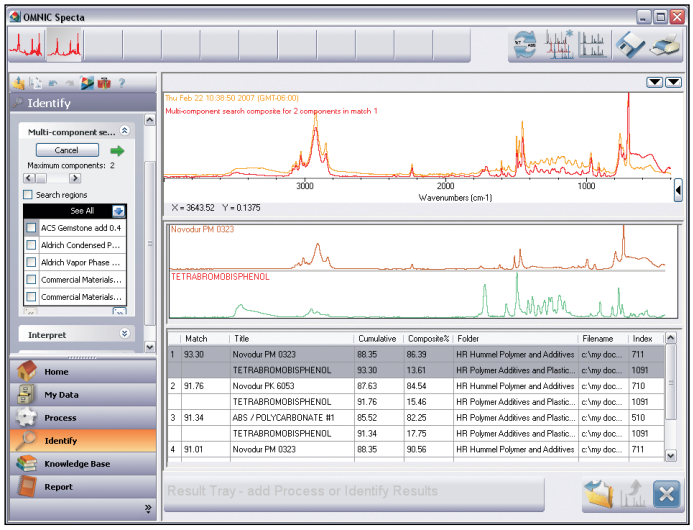

Figure 4: Multi-component result for the same sample as Figure 3. Each top hit combines a base polymer spectrum with TBBP, and the visual agreement is excellent.

A sample compartment TGA accessory was positioned in a Nicolet iS10 spectrometer*, and connected to a TGA furnace. A few micrograms of two samples were run sequentially – an adhesive and an agricultural polymer. Infrared data was collected using the Thermo Scientific™ OMNIC Series™ software. Spectra from temperature points within the OMNIC Series files were exported to OMNIC Specta; no further processing was done.

The extracted spectra and results of the multi-component searches are shown in Figures 5 and 6. The adhesive is emitting four or more gases at once during the heating. OMNIC Specta built a composite that matched the extracted spectrum tightly. In Figure 6, the agricultural polymer is seen to be giving off acetic acid, CO_2 and water. Here, the composite is matching the raw data precisely.

Given that most TGA experiments result in multiple gases being driven off during some portion of the analysis, OMNIC Specta represents a huge leap forward for analytical labs challenged by the deconstruction of these spectra. In these examples, three or more gases are present simultaneously, and OMNIC Specta was completely at ease in extracting the information. As in the previous examples, any user can now analyze these spectra with minimal training and almost no knowledge of infrared searching.

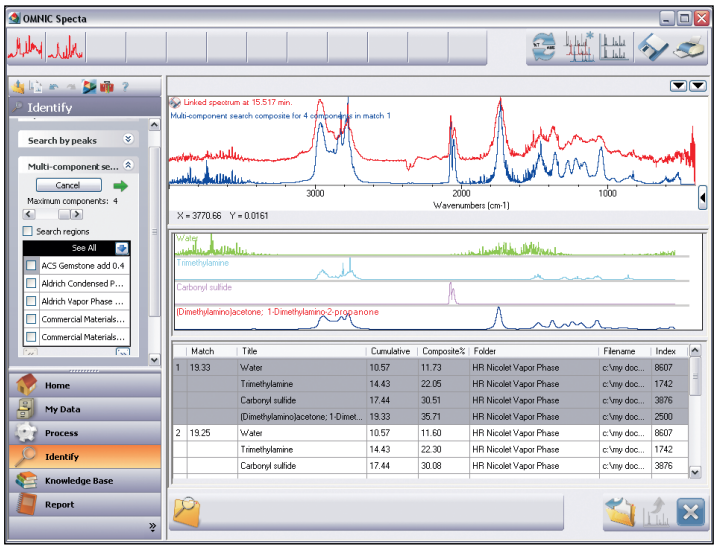

Figure 5: Extracted spectrum from OMNIC Series files for an epoxy examined in the TGA-IR experiment. Note that most of the major features are accounted for.

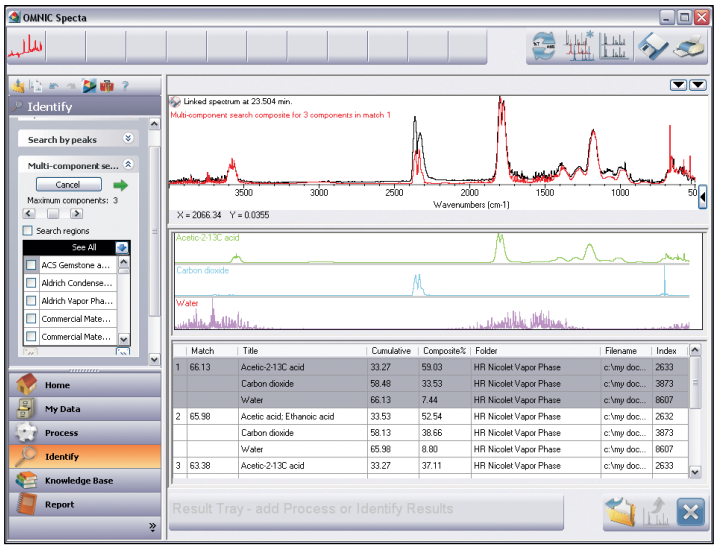

Figure 6: Extracted spectrum for an agricultural polymer in the TGA-IR experiment. The agreement between the multi-component result and the original is spectacular.

Summary

The challenging environment of the analytical laboratory, where samples with unknown composition arrive daily, now has a new ally in the OMNIC Specta spectroscopy software. New tools for organizing and finding data on your computer system and simple processing operations are immensely augmented by the powerful multi-component search algorithm. Short learning curves and consistent operator to operator performance of tasks makes OMNIC Specta accessible and gives great confidence in the results obtained.

References

1. Thermo Scientific Application Note AN50581 "Advanced ATR Correction Algorithm"
2. Waste Electrical and Electronic Equipment Directive and Restriction on Hazardous Substances Directive specify amounts of known toxins that may be present in waste materials.

** With the exception of TGA analysis, experiments can be conducted using the Thermo Scientific Nicolet iS5, iS10 or iS50 FT-IR spectrometer systems. The TGA experiment cannot be run using the Nicolet iS5 spectrometer.*

www.thermoscientific.com

Africa +27 11 822 4120
Australia +61 3 9757 4300
Austria +43 1 333 50 34 0
Belgium +32 53 73 42 41
Canada +1 800 530 8447
China +86 10 8419 3588
Denmark +45 70 23 62 60
Europe-Other +43 1 333 50 34 0
Finland/Norway/Sweden +46 8 556 468 00
France +33 1 60 92 48 00
Germany +49 6103 408 1014
India +91 22 6742 9434
Italy +39 02 950 591
Japan +81 45 453 9100
Latin America +1 561 688 8700
Middle East +43 1 333 50 34 0
Netherlands +31 76 579 55 55
New Zealand +64 9 980 6700
Russia/CIS +43 1 333 50 34 0
Spain +34 914 845 965
Switzerland +41 61 716 77 00
UK +44 1442 233555
USA +1 800 532 4752

Thermo SCIENTIFIC
Part of Thermo Fisher Scientific

TN51506_E 03/13M