Application Note 51773

FT-IR Contaminant Identification using OMNIC Specta Software

Key Words
OMNIC Specta, Contaminant ID, FT-IR, Infrared, Phthalates, Quality Control

Introduction

Many industrial laboratories specialize in determining if a material meets specifications. Materials that pass are deemed appropriate for use in production, or ready for shipment. Materials that fail are generally quarantined for further analysis to determine the source of the failure. Determining the cause of a materials failure is time-critical, since a failure can result in out-of-spec products, which may lead to shipment holds, production holds or worse yet, product recalls.

Infrared (IR) spectroscopy is an ideal analytical tool for both routine quality control (QC) analysis to verify if materials meet specification and analytical investigations to identify the cause of failures when they occur. The utility of infrared for these purposes arises from the simplicity of sample analysis and data acquisition, coupled with the information-rich spectra that it provides.

Thermo Scientific™ OMNIC™ spectroscopy software supplies tools like System Performance Verification to ensure the spectrometer is running properly and that audit trails are complete. Focusing on QC material verification, the Thermo Scientific™ QCheck™ algorithm helps prevent shipment or use of out of specification materials based on the characterization of a unique spectral fingerprint for the known, good material.

When QCheck flags a material as failing, a rapid investigation to identify the source of the failure is essential, as noted earlier. IR has significant utility because it supplies a tremendous amount of information for identifying contaminants. However, sometimes the richness of information contained in the spectra can become a burden. The identification of spectral contributions from unexpected components at low level may be difficult in the presence of known components.

Thermo Scientific™ OMNIC Specta™ software provides a unique and powerful tool to assist with this analysis – Contaminant Search. Contaminant Search complements the breakthrough Multi-Component Search feature in OMNIC Specta by framing the analytical workflow in the specific language of the contamination problem. The key difference is that the main composition of the material is known. Having this information allows the analysis to focus only on unexpected constituents in the material using an automated process.

The traditional process for performing contaminant analysis is spectral subtraction. While useful, spectral subtraction has limitations. First, the presence of totally absorbing peaks and other spectral effects lead to distortions in the subtraction result that complicate spectral identification. In addition, the variability in the subtraction process from user to user can lead to ambiguity in search results based on differences in the resulting spectra. OMNIC Specta performs a concerted search analysis on the sample, using a supplied "pure component" spectrum as one input and a set (one or more libraries) of potential contaminants as the second. This provides a consistent method, with no variation between users, permitting SOPs to be simplified and expedited.

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Materials under regulatory control provide an excellent example. The "contaminant" in this case may be a regulated additive. Children's plastic toys using phthalates as plasticizers (to make the toy pliable) have recently come under regulatory scrutiny due to adverse health affects and are banned in certain countries. Similar regulations apply to flame retardant additives in electronic equipment (WEEE/RoHS) and bis-phenols in general. The most basic situation involves contamination of an industrial process from environmental sources, such as hair, dust, or particles from processing equipment.

Microscopy provides one avenue for contaminant analysis, where spatial resolution permits acquisition of pure spectra from different locations in the sample. However, homogeneous mixtures, such as plasticizer additives or flame retardants, are not spatially separable. There are also times when microscopy would be inconvenient, such as in bulk powdered samples, or is simply not available. In these cases, the IR spectrum represents the co-added spectra from individual components in the sample, and the key part of the analysis lies with untangling the resultant spectrum. This is the core strength of OMNIC Specta.

Experimental

Samples were obtained from various sources. The spectrum from the aluminum can residue was collected using a Thermo Scientific™ Nicolet™ iS™50 FT-IR spectrometer and a Thermo Scientific™ Smart Orbit™ or Smart iTR™ diamond ATR device. IR spectra of pure reference materials (PVC, phthalate, EVA standards) were collected and stored for use in searching. Prior to searching against commercial (transmission) libraries, OMNIC Specta Advanced ATR Correction was applied to permit direct comparison.

Collection of spectra from reference materials is discussed in the Results. Commercial libraries related to the study were also accessed, due to the unknown character of the contaminant in some cases.

Results

The key to any successful contaminant analysis will lie with the quality of the reference spectrum. Advanced ATR Correction allows users of OMNIC Specta to access databases collected using different ATR crystals or transmission, interchangeably. Thus, existing libraries based on transmission do not need to be replaced when ATR is used – the program can easily move between the two. However, the best procedure in an industrial situation will involve collection of data from verified pure materials using the same methodology as used for the questionable samples. These spectra will then feed both OMNIC QCheck and OMNIC Specta Contaminant Search algorithms. The fact that reference data are collected on the same spectrometer, in the same lab, using the same accessory means the information will be highly similar to the spectrum of the contaminated material, making for the ideal comparison scenario.

Visual confirmation of search results is another strong point of the OMNIC Specta interface. Figure 1 shows a contaminant search result with two different viewing options. The "residual" is the portion of the original spectrum not accounted for by the reference material. In the overlay view, the residual and the components which the algorithm determines are present are shown on a common scale, with the components scaled according to their contribution to the spectrum. The offset view is similar, except that the spectra are spread out.

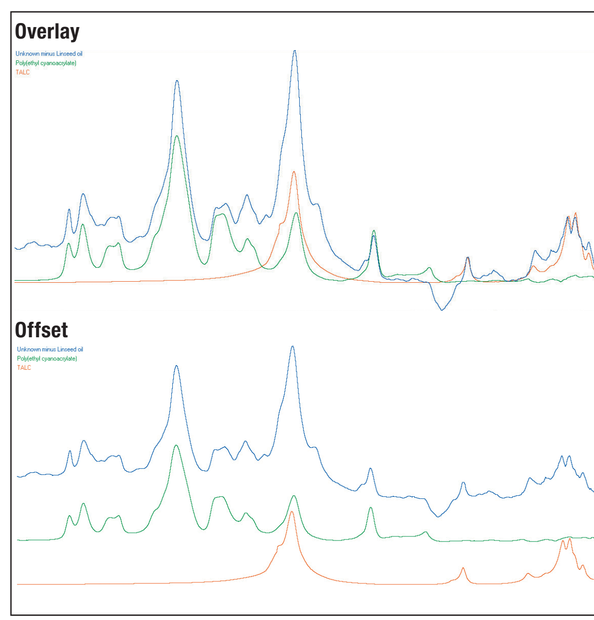
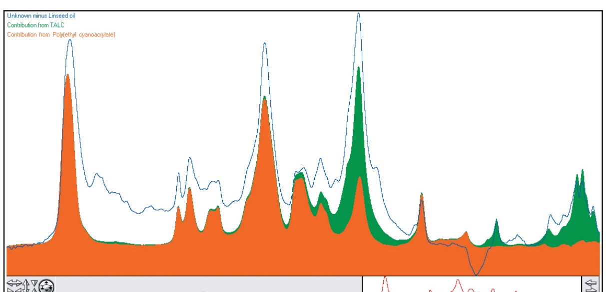


Figure 1

Visual confirmation of search results is another strong point of the OMNIC Specta interface.

Terrain View, shown in Figure 2 for the analysis of an aluminum can, provides a unique way to visualize all of the information in one plot. The residual spectrum is shown as a line overlaying the entire plot (unknown minus known). The colored layers represent the contributions of the successive components. For instance, with two components as shown here, the bottom color represents the contribution of the component with the largest magnitude contribution to the spectrum. The next layer represents the additional contribution from the second component, lying directly atop the first. The envelope (top edge) of the topmost filled layer is the composite spectrum, composed of the two library spectra that best match the residual. The Terrain View allows the user to visually assess how the individual components identified in the unknown spectrum contribute to explain its features.



	Match	Title	Cumulative	Composite%	Folder	Filename	Index
1	85.66	*Linseed oil*	*52.91*	*39.39*	*Known Component*	*c:\my documents\o...*	*703*
		Poly(ethyl cyanoacrylate)	79.31	36.65	HR Hummel Polymer and Additives	c:\my documents\o...	895
		TALC	85.66	23.95	HR Inorganics	c:\my documents\o...	518

Figure 2

The Case of the Stuck Aluminum Can

In this study, a beverage company was accused by a customer of damaging their furniture. The can, when placed on a table top, had left an indelible ring. A sample was obtained by scraping some residue off the base of the can. This scraping was placed directly onto the diamond ATR crystal, and the resulting spectrum is seen at the top of Figure 2.

A standard search indicated the majority of the substance was similar to linseed oil. This was easily explained as arising from oils used in furniture polish. However, there were clearly other components present.

The spectrum was subjected to the OMNIC Specta Contaminant Search, with linseed oil being selected as the "known component" (reference material). Commercial libraries were chosen for the search (Hummel Polymers and Additives plus Inorganics). The Contaminant Search result is shown in the lower portion of Figure 2.

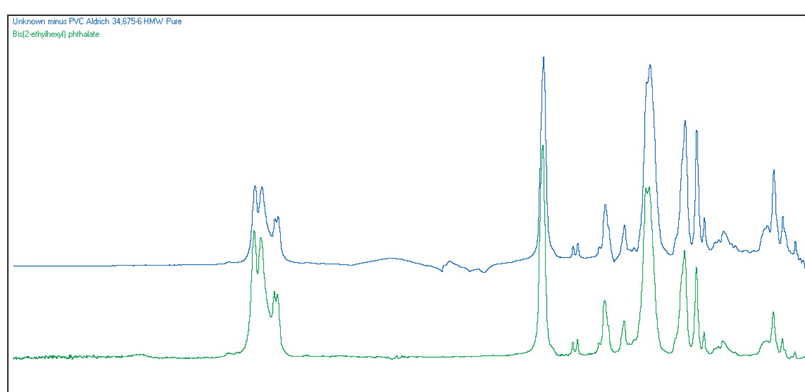
The agreement of the composite spectrum with the original spectrum is excellent. Talc was one of the items identified, a common household material used in foot and baby powders. The story was completed by the third component identified: cyanoacrylate, the key ingredient in super glue. Apparently, the aluminum can had become the focus of a practical joke. Someone had glued it to the table, but the oil allowed the can to release from the surface. In less than two minutes, the problem was solved – from obtaining the can to getting the correct answer.

Plasticizers in PVC

Phthalate additives are commonly used in PVC (polyvinyl chloride) based plastics to provide flexibility. The concentration used depends upon the flexibility desired, but can be quite high. Recent regulatory requirements have made the presence of phthalates a concern to importers, especially of children's toys. In fact certain phthalates have been banned from use in toys due to their strong effect on developing children.

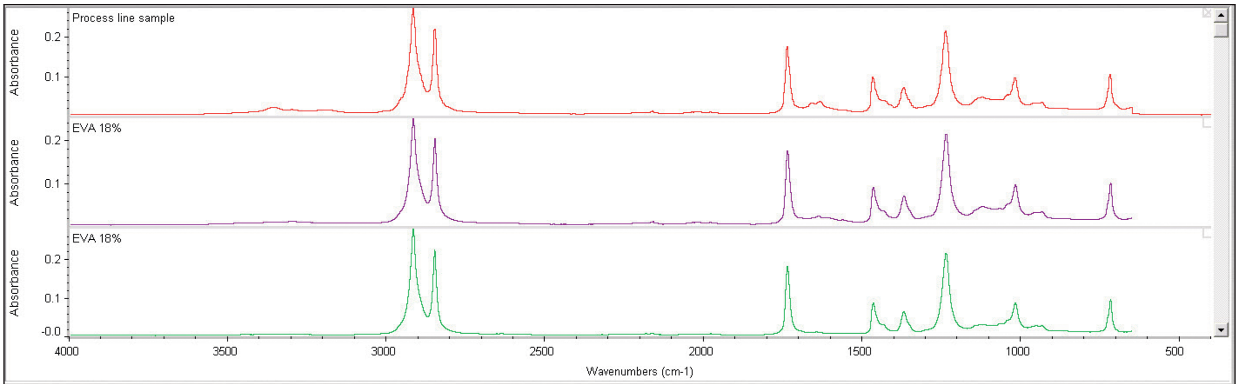
Many PVC samples contain phthalates at some level – pure PVC is actually rather rare. Commercial libraries may contain many PVC spectra, but these can show signs of phthalate additives already. Some care must be exercised in selection of the "pure" PVC spectrum. For this study, we obtained pure samples of PVC powder and key phthalate additives. Spectra were collected and grouped for use as a reference library.

The top of Figure 3 shows the spectrum of a PVC material. Using Contaminant Search with a pure PVC spectrum as the known component and the Hummel Polymers and Plasticizers library for the search yields the result shown at the bottom. Clearly, OMNIC Specta has pulled out the specific phthalate, not just the general class of material, in one simple step.



	Match	Title	Cumulative	Composite%	Folder	Filename	Index
1	93.83	*PVC Aldrich 34,675-6 HMW Pure*	*57.71*	*94.69*	*Known Component*	*c:\my documents\o...*	
		Bis(2-ethylhexyl) phthalate	93.83	5.31	HR Hummel Polymer and Additives	c:\my documents\o...	1198

Figure 3



	Correlation (High Sensitivity)	Reference Title	QCheck Region	Pass/Fail
1	0.9601	EVA 18%	4000.0-650.0	Fail
2	0.9461	EVA 18%	4000.0-650.0	Fail
3	0.8802	EVA 15%	4000.0-650.0	Fail
4	0.8123	EVA 24%	4000.0-650.0	Fail
5	0.8096	EVA 24%	4000.0-650.0	Fail
6	0.8052	EVA 15%	4000.0-650.0	Fail
7	0.6111	EVA 28%	4000.0-650.0	Fail
8	0.6054	EVA 28%	4000.0-650.0	Fail
9	0.4449	EVA 5%	4000.0-650.0	Fail
10	0.4410	EVA 5%	4000.0-650.0	Fail

Figure 4

Contamination in an Industrial Process: EVA

A comprehensive demonstration of the tools from the OMNIC software suite is shown in the final example. A production line was scheduled to make (poly) ethylene vinyl acetate (EVA) with 18% EVA. The production requirements were very strict, so a high sensitivity quality check using the OMNIC QCheck feature with a threshold of 0.98 was done. The material in Figure 4 failed – narrowly – with 18% EVA being the closest match and the expected result.

The spectrum was sent directly to OMNIC Specta from OMNIC and a simple spectral search was performed. The top hit, 18% EVA, was the same spectrum that QCheck had identified as the main component. The high hit metric – 99.13 – could lead to an incorrect conclusion that the search is done, if not for both the QCheck result (fail) and visual overlay comparison of the spectra. A right click on the top hit result placed the 18% EVA spectrum into the Contaminant Search window. Using the Polymer Additives and Plasticizers library for the search, a result came back in seconds. Note that QCheck had already told us what reference spectrum to use, which search merely confirmed – we could have skipped the search and gone directly to Contaminant Search with this spectrum specified as the known component.

The result is shown in Figure 5, with a visually excellent agreement. The contaminant was erucylamide (erucamide), a material from the general class of slip-aids commonly placed on plastic bags. This could be confirmed by right clicking on the result and performing a web search. The search shows the content of this contaminant to have been at a very low level – later verified to be about 0.8% – pointing to the sensitivity of the Contaminant Search algorithm.

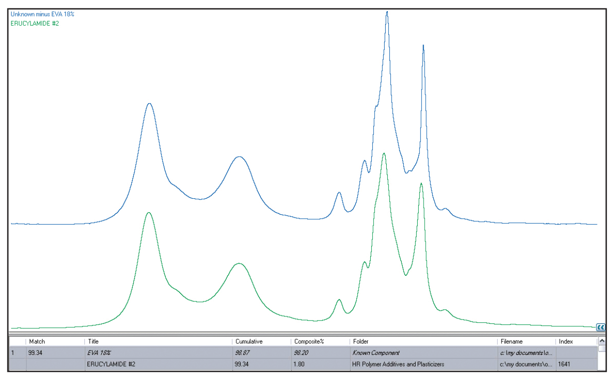


Figure 5

Conclusion

OMNIC Specta provides an incisive tool for identification of contaminations in materials. Combining its powerful algorithm with an extensive collection of Thermo Scientific infrared spectral libraries makes this come alive for work in QC, analytical service and material analysis laboratories. The comprehensive visualization options give the user the insights needed to support their findings and prove their case.

** Experiment can be conducted using the Thermo Scientific Nicolet iS5, iS10 or iS50 FT-IR spectrometer systems.*

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