

# Mechanical fluid analysis for particulates via FTIR microscopy

**Key words:** Automotive, Infrared, Filters, FTIR Microscopy, Particulates, QA/QC

## Abstract

Moving parts, from turbojet engines to artificial joints, produce signs of wear often in the form of abraded particulates which are carried away by fluids like lubricants or coolants. An analysis of the number, size and identity of particulates provides a measure of both severity and origin of wear. FTIR microscopy is very well suited to the analysis of particulates originating from seals, gaskets, adhesives and environmental sources. The particulates are typically 20 microns or larger in size, making them easily trapped on filters. We show how a simple point-and-shoot FTIR microscope aids in the rapid identification of filtered particulates.

## Introduction

Modern living relies on moving machinery of many types. Friction and abrasion of adjacent materials produces particulates. The liquids surrounding these moving parts – lubricants, coolants, fuels or even biological fluids – carry these particulates and may deposit them in critical locations. Filters used both during operation and after fluid extraction (such as a coolant change) trap the particles. The characteristics of the particulates – size, number and composition – provide critical diagnostic information regarding component wear.



Gaskets, o-rings and other polymeric materials are critical in many automobile moving components. The particles generated from wear can be trapped either by in-line filters (like an oil filter) or off-line filter after extraction. The trapped material can often be easily analyzed using FTIR microscopy, although the lack of affordable equipment and expert users may require sending the filters to external labs, which is expensive and time consuming.

We demonstrate here the use of the low-cost, simple-to-operate Thermo Scientific™ Nicolet™ iN™5 FTIR microscope for analysis of particulates. The visual capabilities and straightforward infrared data collection of the Nicolet iN5 microscope mean that even an inexperienced operator can locate and identify particulates on filters in minutes without sample preparation or extensive training. Further, the Nicolet iN5 microscope has no motorized parts to fail, ensuring a long lifetime.

## Experimental

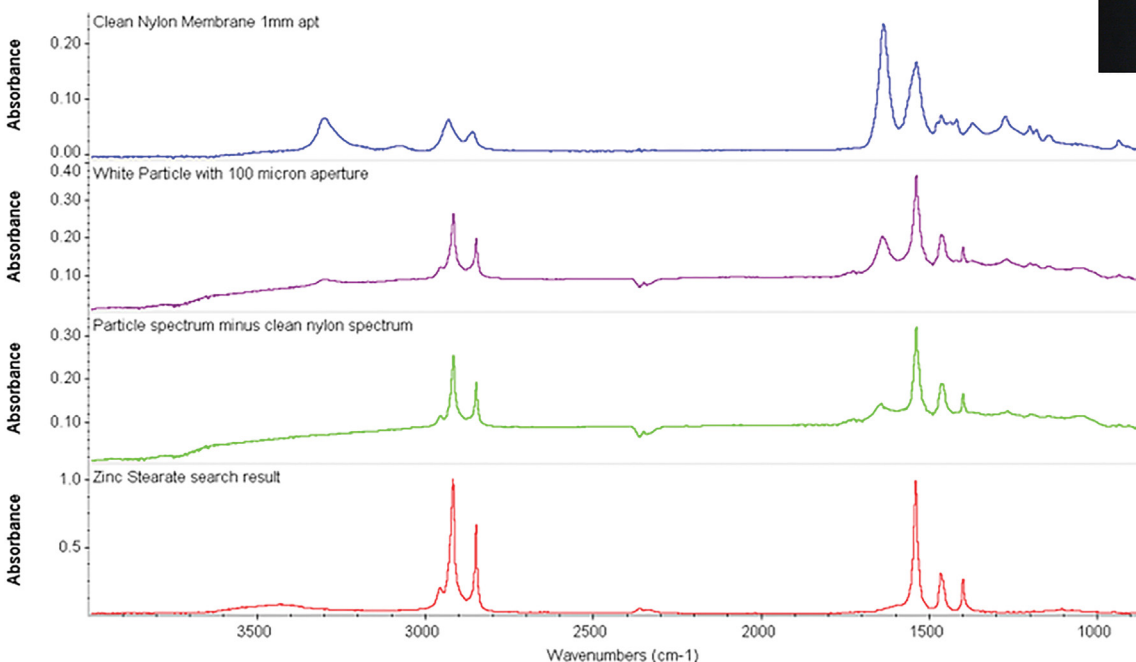
A Nicolet iN5 FTIR microscope connected to a Thermo Scientific™ Nicolet™ iS™10 FTIR spectrometer, shown in figure 1, was used for the analysis. The system included a Germanium 'tip' attenuated total reflection (ATR) sampling device which permits touch-and-collect simplicity. The concepts behind ATR data collection are presented elsewhere.<sup>1</sup>

Cooling water, transmission fluid and fluid extracted from a shock absorber were filtered (off-line). The filters, made from a soft polymer, were air-dried, then mounted on glass slides using double-stick tape near the edges to hold them flat – no other sample preparation was needed. A target particle was located and centered in the video image. A fixed, 1 mm aperture which yielded a 25 micron effective sample aperture was used.<sup>2</sup>



**Figure 1:** The Nicolet iN5 microscope attached to a iS10 FTIR spectrometer.

The Ge-tip ATR was inserted and a background spectrum of the clean ATR crystal was acquired using the Thermo Scientific™ OMNIC™ spectroscopy software. Using the OMNIC preview-mode, the microscope stage was slowly raised. The appearance of the spectrum and color change in the LED pressure indicator provided confirmation of readiness for data collection.



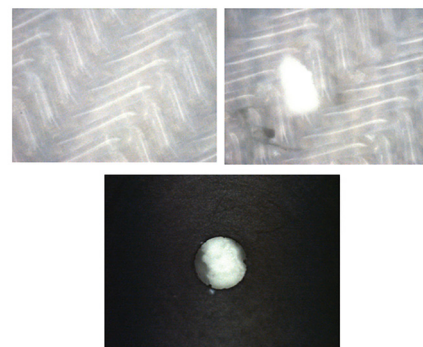
**Figure 3:** Spectra collected from locations shown in Figure 2. Top: bare filter. Second: white particle. Third: subtraction result. Fourth: Search result for subtracted spectrum.

The high throughput of the Nicolet iN5 microscope and the excellent signal-to-noise of the Nicolet iS10 spectrometer enabled data collection to complete within seconds, sometimes even with a single scan. Processing and analysis using OMNIC or Thermo Scientific™ OMNIC Specta™ software was also quick, so the total time for this analysis (loading sample to identification) was less than 3 minutes in most cases.

## Results

The first filter came from engine coolant. The visual image of a clean filter, a particulate and particulate centered in the 100 micron aperture are shown in Figure 2. The spectrum from the clean filter, shown at the top of Figure 3, indicates these are made from nylon (search match not shown). The second spectrum was collected from the particle. There is some evidence of nylon bands in this spectrum. Spectral subtraction removes the small nylon bands, yielding the third spectrum. Searching yields a strong match to Zinc Stearate, shown at the bottom, which is common mold-release agent used in plastic parts production. This was likely 'washed' off the surface of a component by the heated fluid.

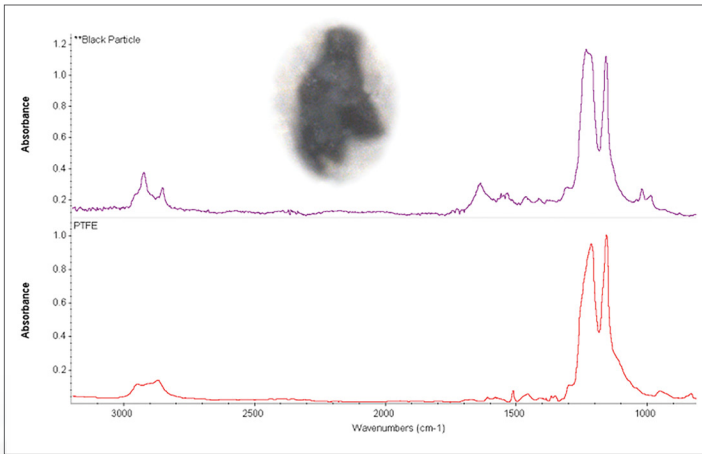
**Figure 2:** Top left: image of the bare filter. Top right: a single particle on the filter. Bottom: image through the aperture.<sup>2</sup>



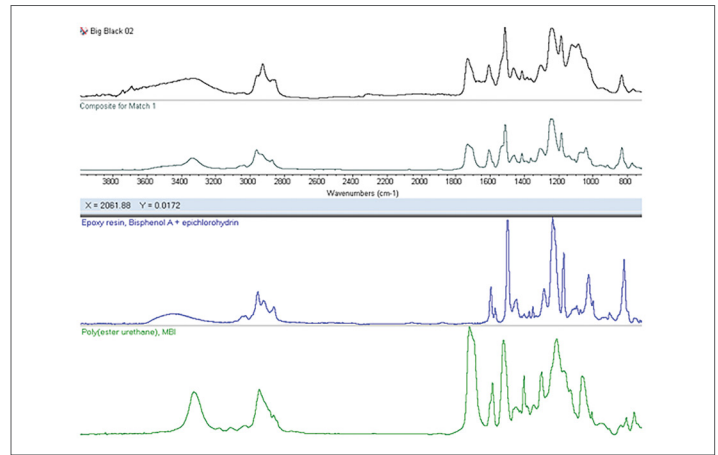
A number of different types of particles were seen when fluid from a shock absorber was filtered. First, a highly irregular black particle which was slightly embedded into the filter is seen in the photo inset in Figure 4. The background signals from the nylon filter are very small, so searching occurred without processing. The analysis yielded PTFE (Teflon™), indicating wear of a sealing ring within the piston of a shock absorber.

While visibly similar, another black particle on this same filter gave a very different result. The spectrum, shown in Figure 5, appeared to be a mixture, so OMNIC Spectra was used for the searching. The results indicate the presence of a phenoxy resin, which is a dispersant material for colloidal suspensions, and poly(ester urethane). A yellow particle on this same filter, seen in Figure 6, appears to be an organic based pigment (paint) chip.

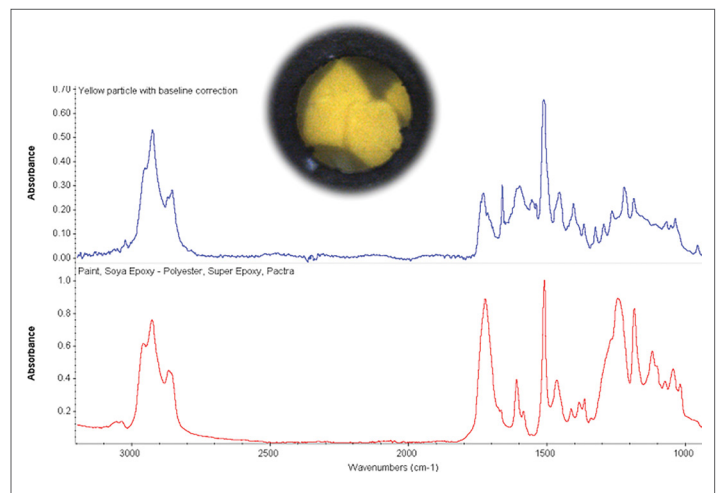
The final particle analyzed showed a more complex mixture spectrum. The image of the bright orange particle and the search results are shown in Figure 7. There is very strong evidence once again for PTFE. The second match is a styrene-butadiene copolymer, likely indicative of wear on a plastic part. The final result is a lubricant common in automotive uses.



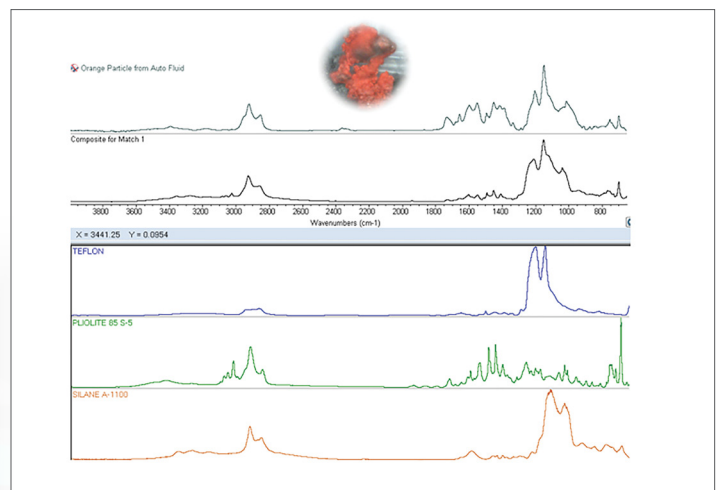
**Figure 4:** Black particle, spectrum and search result showing PTFE.



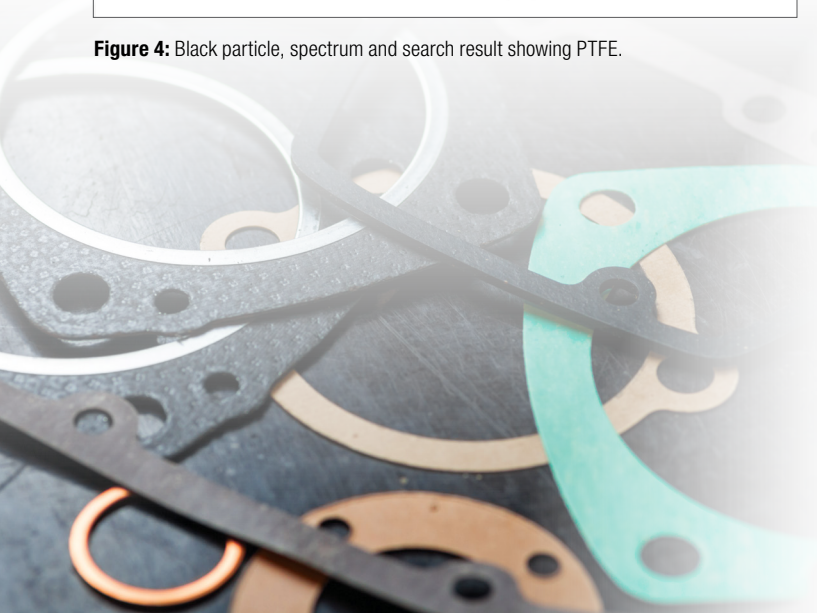
**Figure 5:** Multicomponent search result for a second black particle. Top: raw spectrum. Second: composite or sum of the chosen search matches. Bottom two: Epoxy resin and Poly(ester urethane) matches.



**Figure 6:** Yellow particle, spectrum after baseline correction and search result.



**Figure 7:** Orange particle and three-component search result from OMNIC Spectra.



## Conclusion

The abrasion present with moving parts produces particles. In many cases, these can be analyzed simply with a FTIR microscope. The Thermo Scientific Nicolet iN5 microscope and OMNIC suite of software provides a rapid and simple analysis tool usable with little or no sample preparation. The ability to analyze such materials without shipping samples to testing laboratories can save considerable time and money.

## References

1. Visit the FTIR Spectroscopy Academy for more information at [www.thermofisher.com/FTIRAcademy](http://www.thermofisher.com/FTIRAcademy)
2. The aperture is 1000 microns in diameter. The optics of the Nicolet iN5 are set to 10X, so the visual aperture is reduced to 100 microns. The Germanium ATR also acts like a focusing lens due to its high index of refraction, further reducing the IR aperture by 4X, yielding the final 25 micron aperture setting.

Find out more at [www.thermofisher.com/iN5](http://www.thermofisher.com/iN5)

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