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FTIR Analysis of Samples 12345.001-003

1. Introduction

In this section we include a brief introduction covering sample information and customer requirements.

In this example Swansea Tribology Services received a sample of fibres from the factory's Lube Oil Filters and several reference samples, with the request to identify the origin of the fibres. The samples were allocated lab references as follows:

12345.001 – Fibres from Lube Oil Filters
12345.002 – Example of a Rag in use around the factory
12345.003 – Example of an Oil Absorbent Pad in use around the factory

Microscopic analysis could not definitively identify the source of these fibres, so FTIR analysis of the fibres and reference materials was proposed.

Where necessary, we include photos and micrographs of the samples to aid with understanding of the analysis and show areas sampled.

Figure 1.1 Filter membrane with fibres, sample 12345.001 – Fibres from Lube Oil Filters

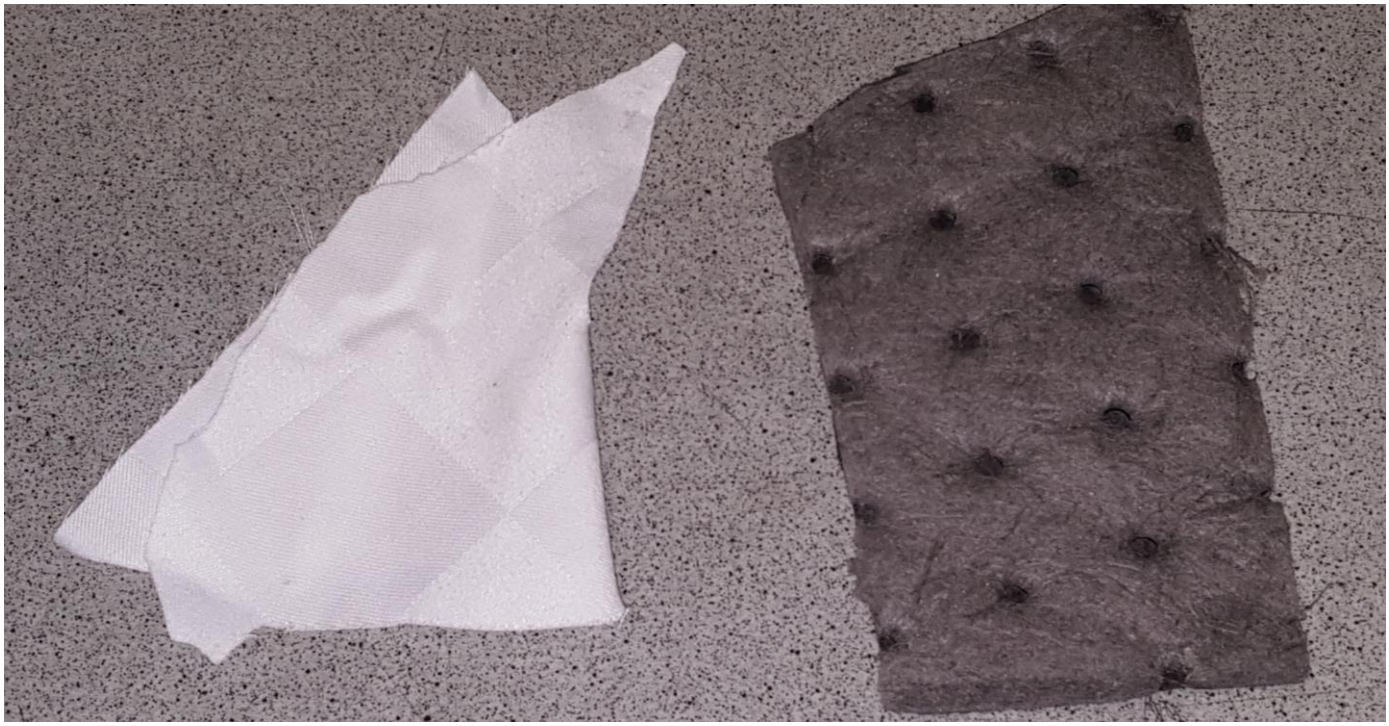

Figure 1.2 Rag and Absorbent Pad, samples 12345.002 and 12345.003

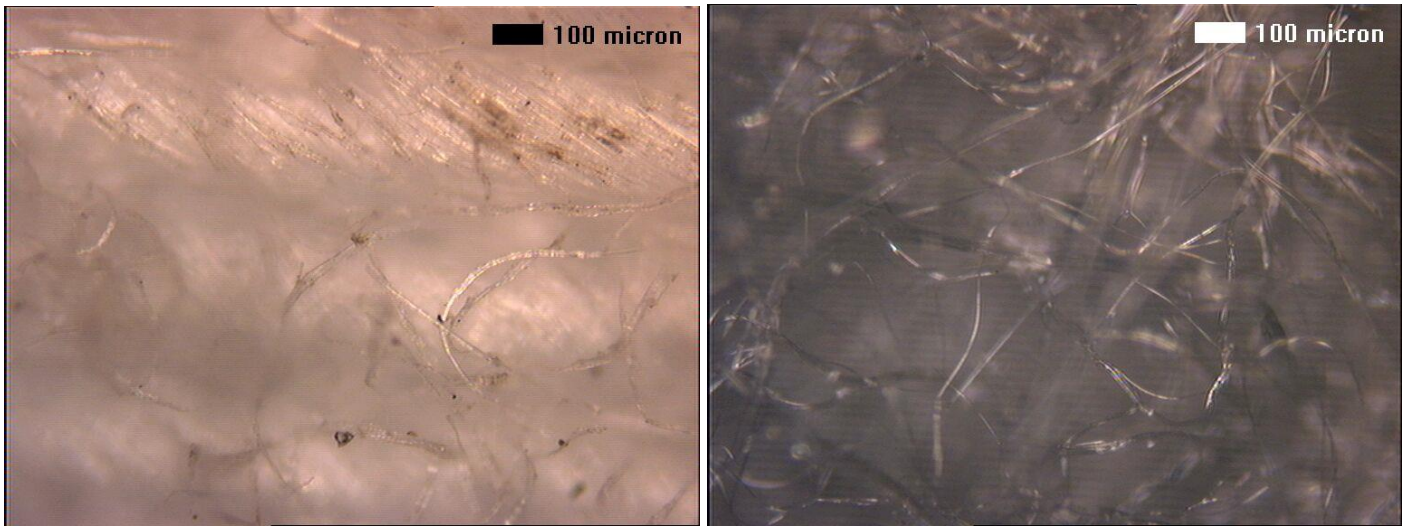

Figure 1.3 Microscopic view of Rag and Absorbent Pad fibres, samples 12345.002 and 12345.003

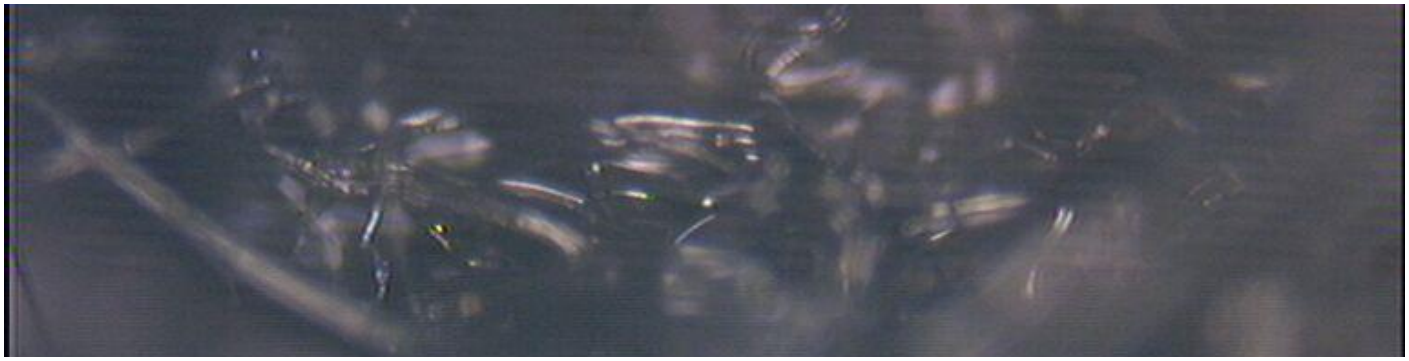

Figure 1.4 Microscopic view of fibres from debris sample, sample 12345.001

2. FT-IR Analysis

Note: *In* ***FTIR Spectroscopy*** *vibrations of molecular bonds in response to infrared excitation are measured, producing an infrared spectrum. Different molecular bonds generate responses at different, often multiple, wavelengths due to the type of the bond and the weights of the constituent atoms. Thus, the peaks of an infrared spectrum can provide information on the molecular bonds present and characterise the compound being measured. The quantity of organic compounds in existance is vast, with many consisting of a complex arrangement of similar molecular building blocks and therefore having many bonds in common. Presence of multiple substances within the sample can complicate the matter further.*

Here follows a summary of how the samples' FTIR spectra have been collected. If additional sample preparation or manipulation was required, this will also be stated here. The main two options are to use a ZnSe transmission cell or a Diamond UATR accessory.

Note: *The ZnSe transmission cell offers better sensitivity and improved definition of trace substances, as the beam pathlength through the sample is increased. This can, however, lead to oversaturation where especially strong peaks are generated. It can only be used for liquids and is not compatible with every type of fluid, as some can attack the cell. It can be damaged by abrasive materials within the fluid and cannot be used for particularly viscous samples. As well as investigative analysis it is also used for routine analysis of lubricating oils and allows for accurate measurement of Soot, Oxidation, Nitration, Sulphation, Water, Anti-wear Additive levels and, where calibration has been performed, Fuel and Glycol content.*

The Diamond UATR accessory collects a reflectance spectrum. The signal does not penetrate very far into the sample, which could lead to a reduction in sensitivity. The UATR accessory can be used for either solid or liquid samples of various composition. Hard materials can be crushed into powder and pressed against the cell to improve contact. This is a very versatile option and is used a lot for non-routine investigative analysis.

Figure 2.1 FTIR Transmission Cell and Perkin Elmer Spectrum Two FTIR Spectrometer with a Diamond UATR Accessory fitted

In this example the samples were analysed using a Perkin Elmer Spectrum Two Infra-Red Spectrometer with a Diamond UATR accessory. Figures 2.2-2.4 show the FTIR spectra thus obtained.

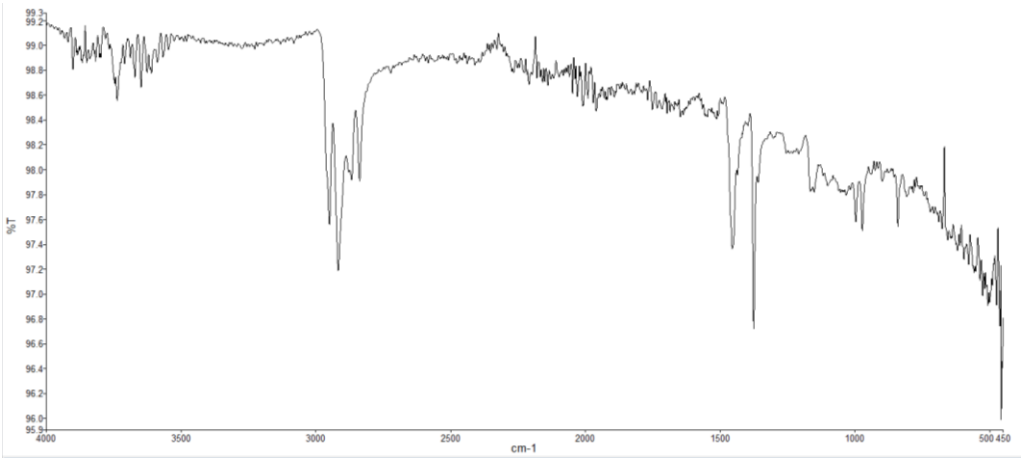

Figure 2.2 FTIR spectrum of fibres from sample 12345.001 – Fibres from Lube Oil Filters

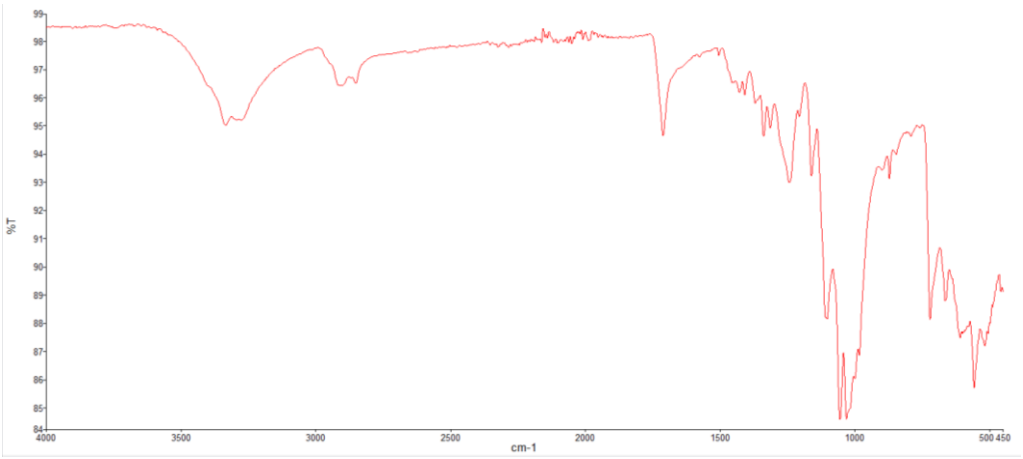

Figure 2.3 FTIR spectrum of sample 12345.002 – Rag

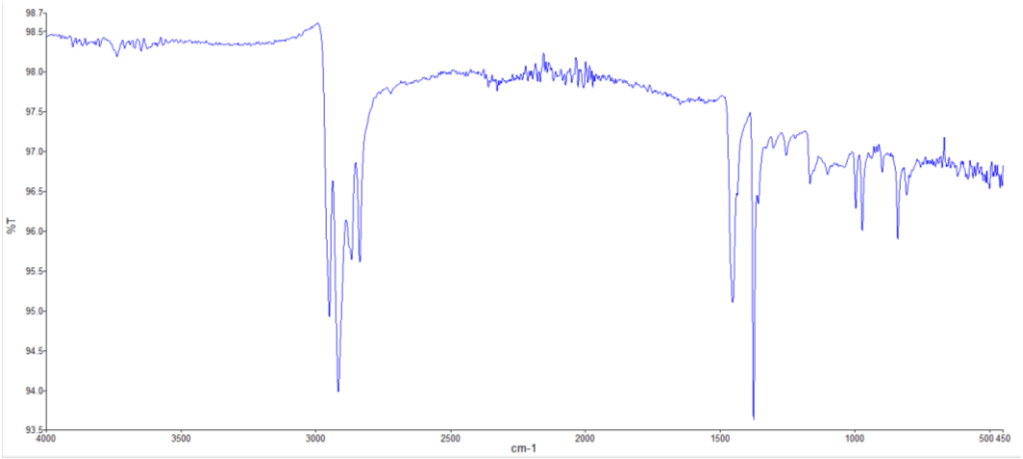

Figure 2.4 FTIR spectrum of sample 12345.003 – Absorbent Pad

The Rag and Absorbent pad fibres appear to have distinct molecular compositions. The unidentified fibres appear to be a direct match to the Absorbent Pad material with a good correlation between the characteristic peaks.

Overlaying spectra helps verify exact alignment. Zooming in on specific wave ranges helps identify matching regions as well as any divergent peaks, see Figures 2.5 and 2.6.

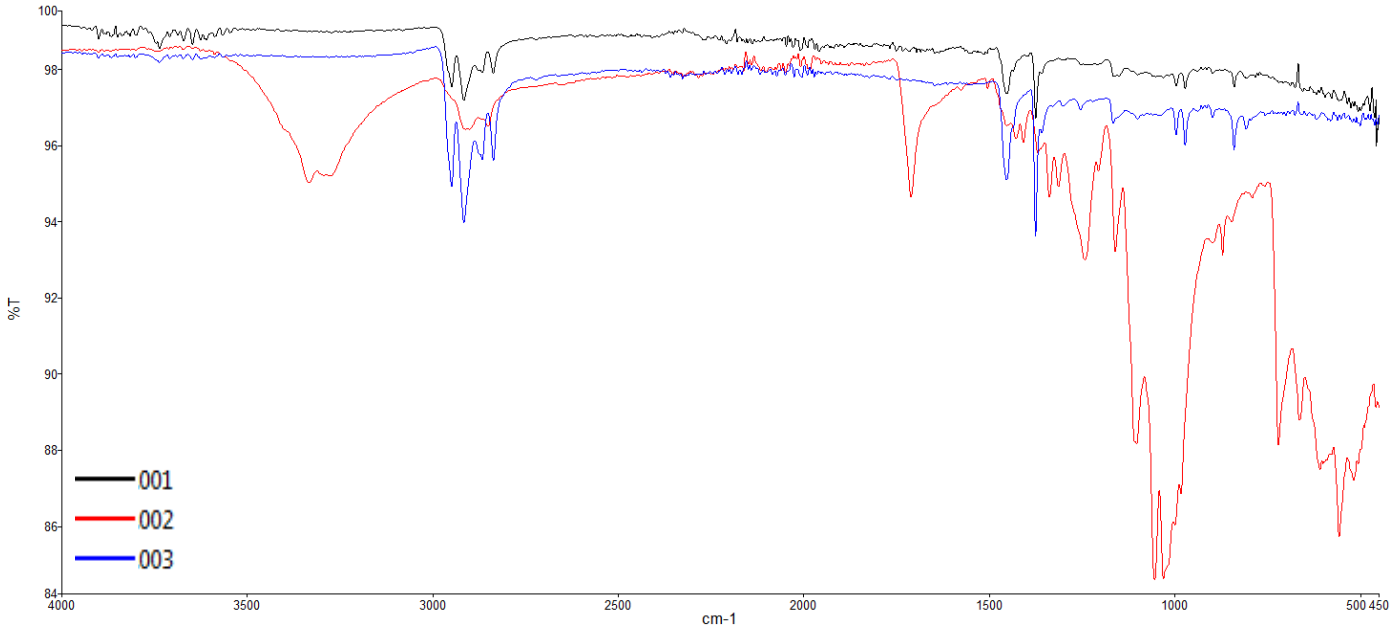

Figure 2.5 FTIR spectra of samples 12345.001-003 – Fibres from Filter Debris, Rag and Absorbent Pad

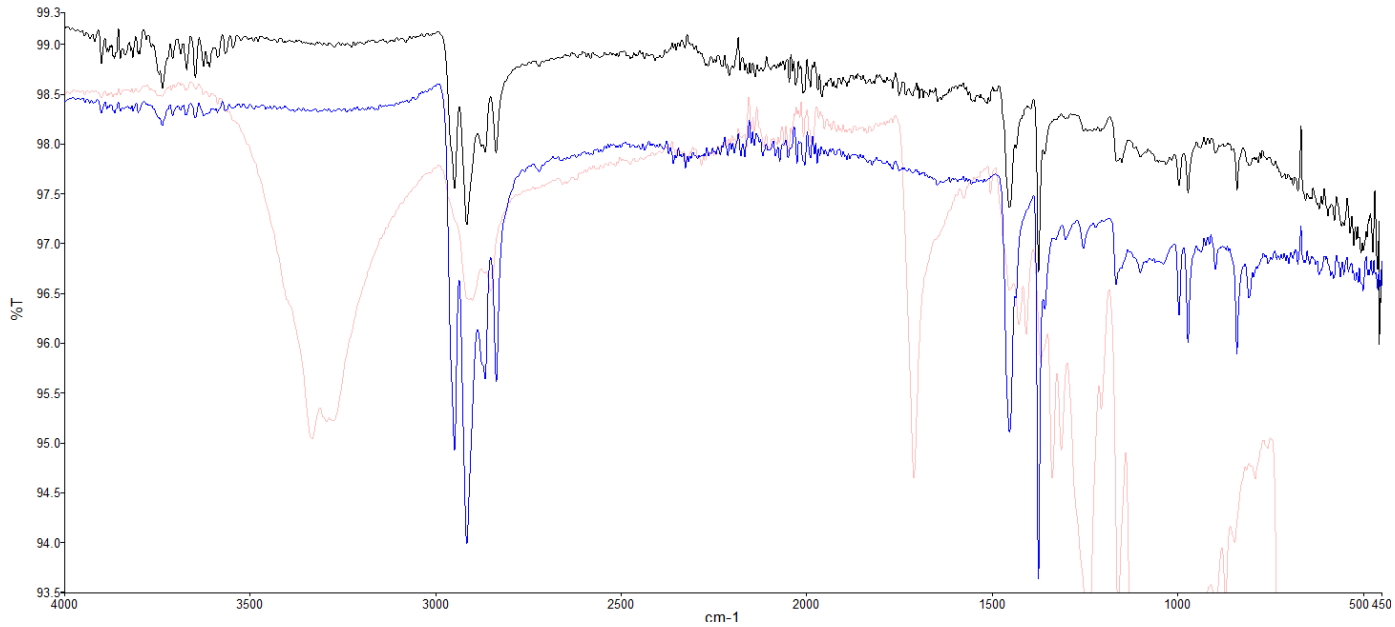

Figure 2.6 FTIR spectra of samples 12345.001 and 12345.003 – Fibres from Filter Debris and Absorbent Pad

In some cases, direct comparison is not sufficient. Where more than one compound has been mixed, spectral subtraction can be used to remove one of the constituent components and review the residual spectrum against candidate contaminants.

In the following example a sample of oily emulsion was to be compared to a range of candidate contaminant samples from around the process. The collected sample presented quite a complex

spectrum (Figure 2.7), however during storage a layer of neat oil has separated within the sample. This layer was also scanned, and the spectrum of the top oily layer can be seen together with two scans of the emulsion in Figure 2.8

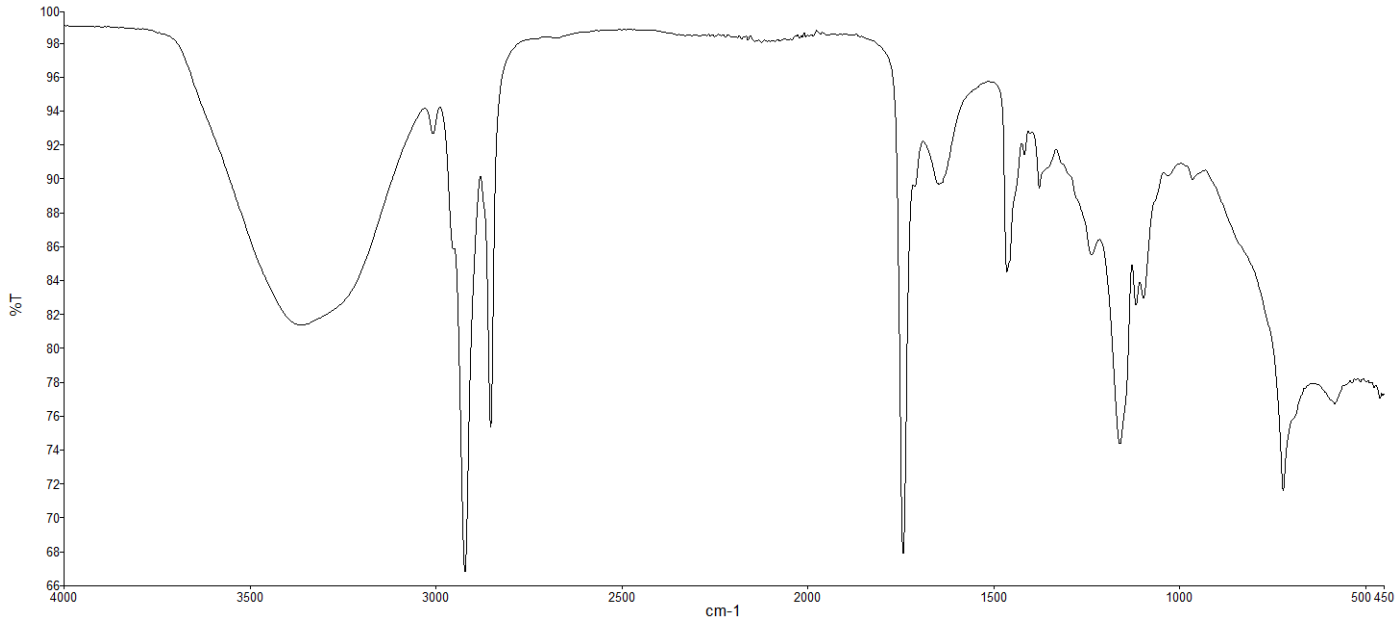

Figure 2.7 FTIR spectra of emulsified sample 56789.001

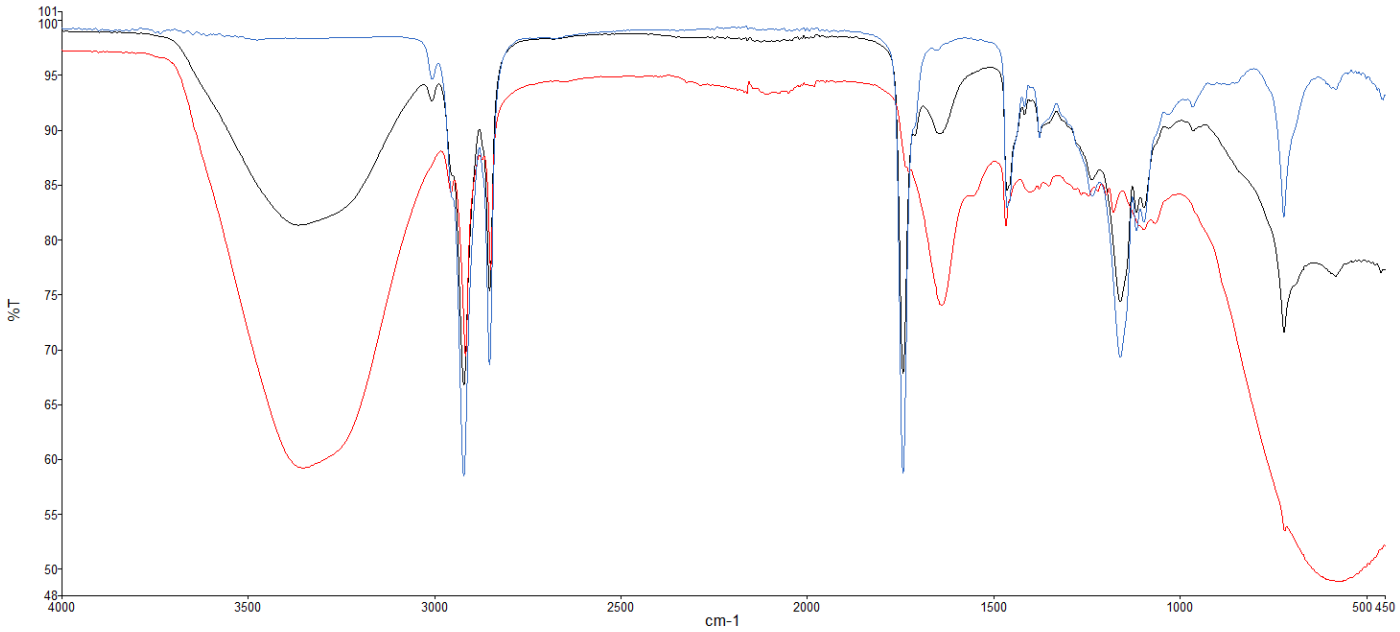

Figure 2.8 FTIR spectra of emulsified sample 56789.001. Top oily layer (blue) and two measurements of the emulsified layer with and without application of pressure against the UATR cell (red and black respectively)

After using a spectral subtraction algorithm, the residual spectrum was found to be virtually identical to a library spectrum of water, with no evidence of any other contaminants.

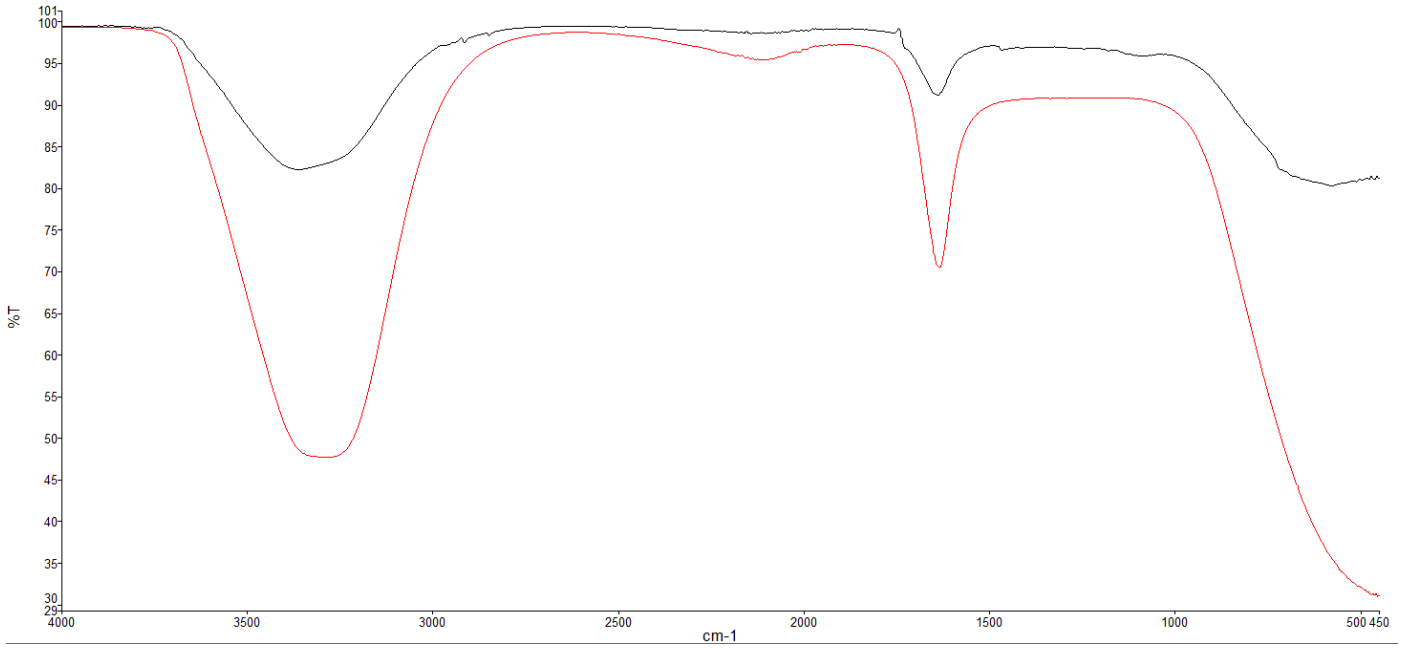

Figure 2.9 FTIR spectra of residual sample 56789.001(black), and distilled water (red)

Where no candidate samples have been provided, a library search can be used to learn more about the compound. In some cases, a match can be found among the previously tested samples. At other times, a generic library can give indication of the likely composition (see figure 2.10). Where necessary scans can be submitted for searches against various other libraries at additional cost.

In the following example the contaminant was narrowed down to an ethylene glycol compound, making identification of the source much easier for the customer.

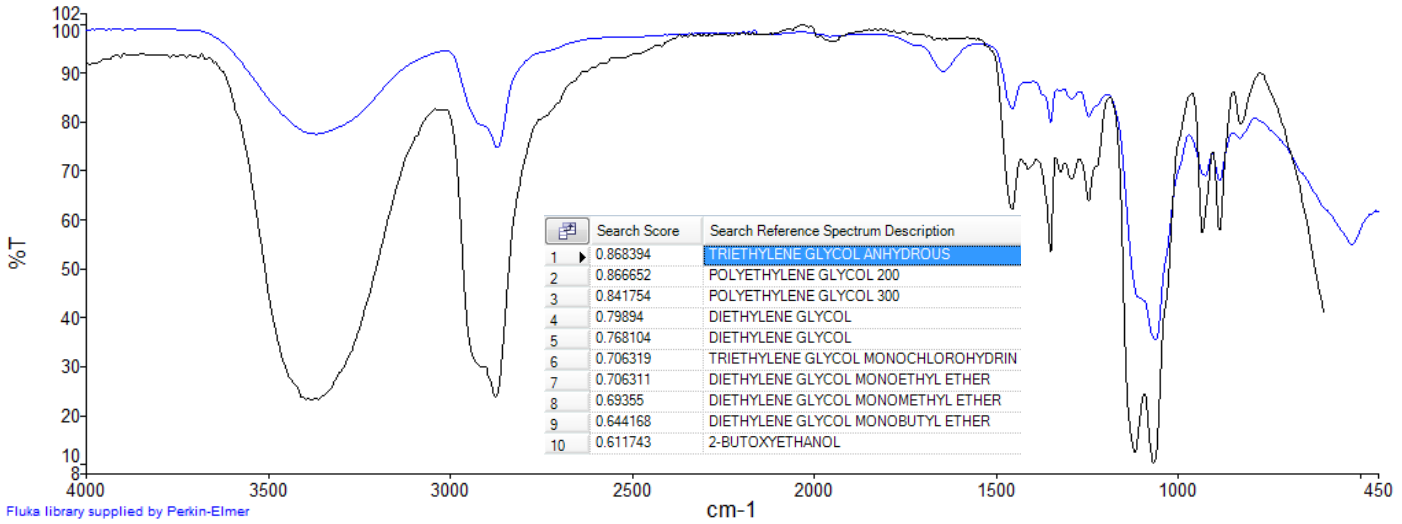

Figure 2.10 A library search suggesting that the sample was consistent with ethylene glycols

If all else fails, looking up individual peaks can provide some information on the likely constituents. This, together with other data available, can help narrow the search to a particular class of compounds or exclude certain candidates.

3. Conclusion

In this section we summarise the conclusions from the above analysis. Where other data is available it may be used to confirm a particular conclusion.

The fibres from the Lube Oil Filter Debris sample appear consistent with the Absorbent Pad material and not with the Rag material.

The emulsion appears to be that of oil and water without significant quantities of any other substances present.

Sample in Figure 2.10 exhibited correlation with a number of ethylene glycol or related compounds.

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