Infrared Detector

FUNCTIONAL GROUP ANALYSIS OF AROMATICS, ESTERS AND ALCOHOLS

Traditional petroleum fluids are very complex. Recent new fuel sources only add to the complexity of fuel and petroleum characterization. Current gas phase analytical methods for petroleum fluids emphasize methods that expect non-polar analytes in the samples. These non-polar analytes mainly consist of a wide range of hydrocarbons. Exceptions include the analysis of oxygenates in finished fuels in which a defined mixture of alcohols may be added to the final petroleum fuel at the final blending stage.

New fuel source materials introduce a wide range of new analytes into the analysis and characterization of source fuel and mixed petroleum samples. These new fuel related analytes include, new functional groups, as well as more polar chemistry. While this introduces new challenges to the established petroleum analytical techniques, it also allows new opportunities for quick analyze and characterization of these new samples.

New fuel source materials are still developing and they include a wide range of plant based or refuse based materials. These initial source materials are processed to generate a stream that can be further modified and refined for fuel use. Both these source materials and the processing techniques require analytical characterization to assist in optimization. In this application, the ASAP IRD 3, a gas phase infrared detector for GC eluents, is used to provide selectivity and identification in complex samples. The IRD and accompanying software is used with an Agilent 7890 GC with split injection and capillary separations prior to IRD detection.

Here is a separation of a complex new source fuel mixture by GC-IRD. First the Total Response Chromatogram (TRC) is shown with response at all wavelengths. This essentially shows the entire chromatogram without selectivity.

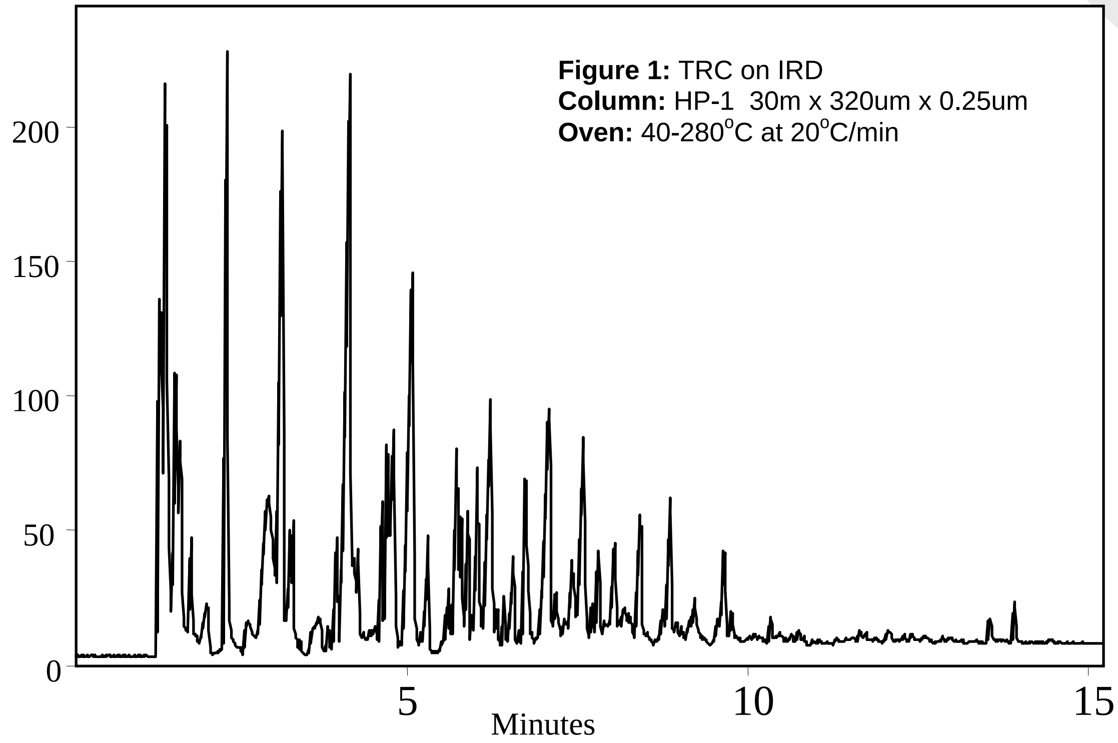

Figure 1: TRC on IRD
Column: HP-1 30m x 320um x 0.25um
Oven: 40-280°C at 20°C/min

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We can now look at this same complex sample with functional group selectivity. Here is a SWC chromatogram at 1750 wavenumbers and now we only see the ester and carboxylic acid analytes (carbonyl groups) in this sample:

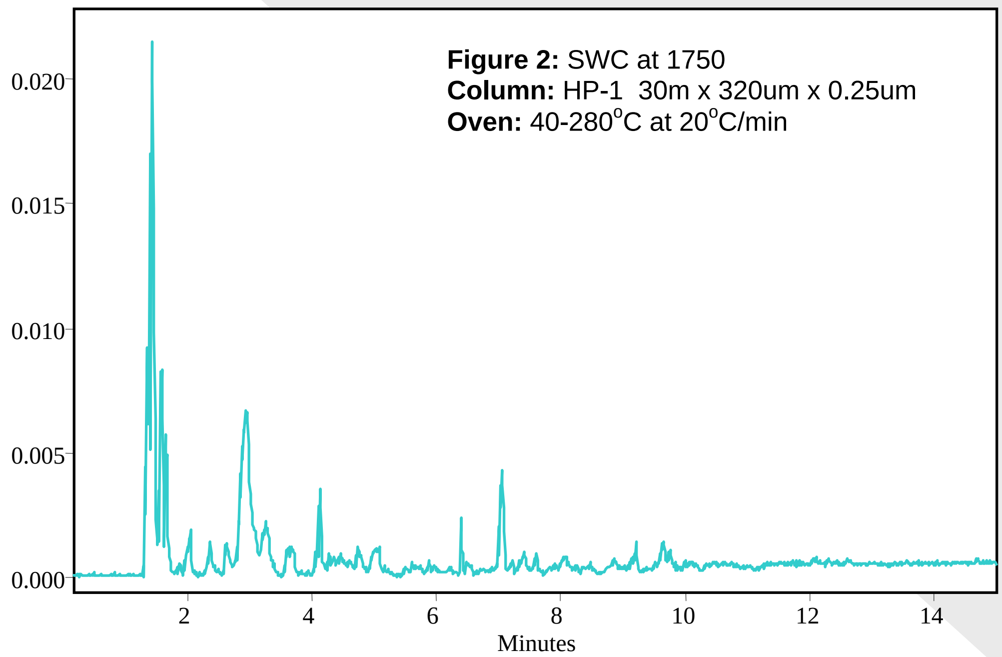

Figure 2: SWC at 1750
Column: HP-1 30m x 320um x 0.25um
Oven: 40-280°C at 20°C/min

This complex sample also contains many alcohols. Here is a SWC chromatogram at 3600 wavenumbers and we see all of the alcohols, phenols and free acids (-OH group) in this sample

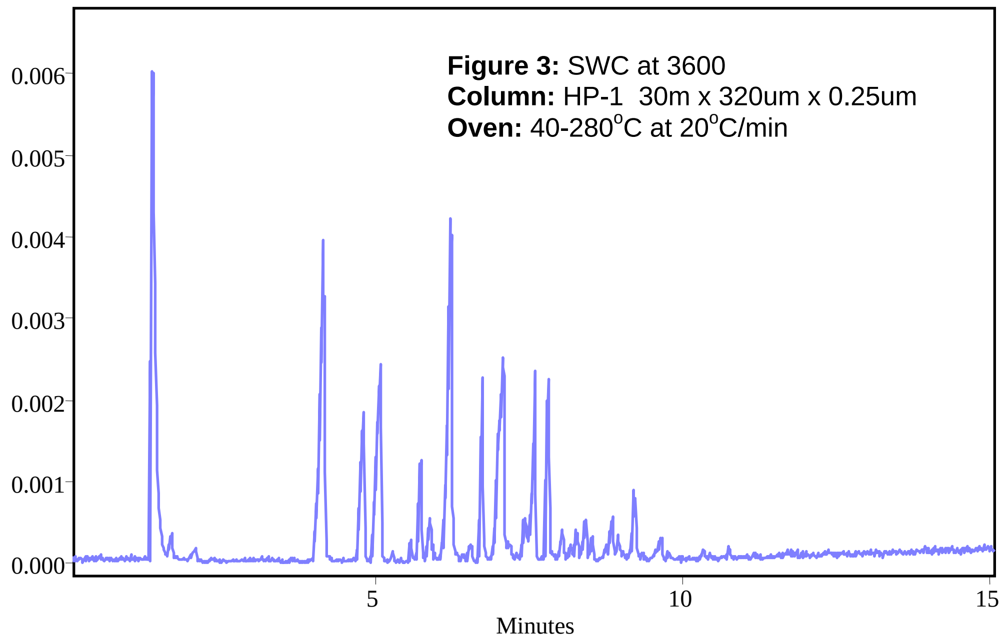

Figure 3: SWC at 3600
Column: HP-1 30m x 320um x 0.25um
Oven: 40-280°C at 20°C/min

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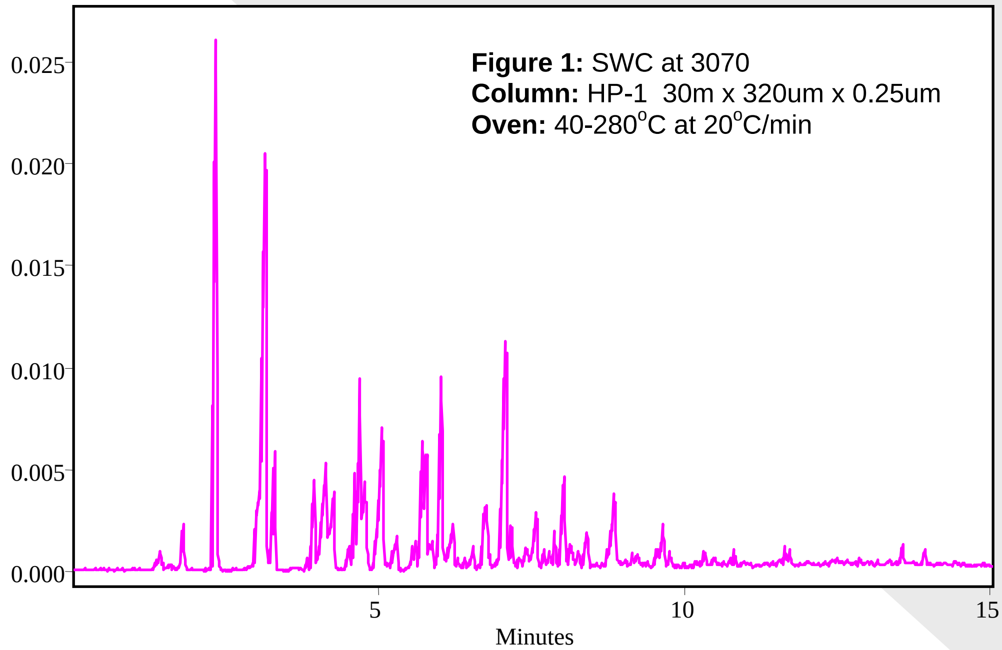
This complex sample also contains many aromatic analytes. Here is a SWC chromatogram at 3070 wavenumbers and we see the aromatic analytes in this sample:



These three selective SWC chromatograms quickly show examples of a range of functional group distributions for this complex sample. Now we can identify individual peaks or even unresolved peaks with the IR vapor phase libraries. Here are some examples. The software is simple to use and involves clicking on a peak of interest to show the subtracted spectra. The reference can be easily adjusted by the user. In these examples, the top spectra in each pair is the best library search match for the bottom analyte spectra taken from the sample chromatogram

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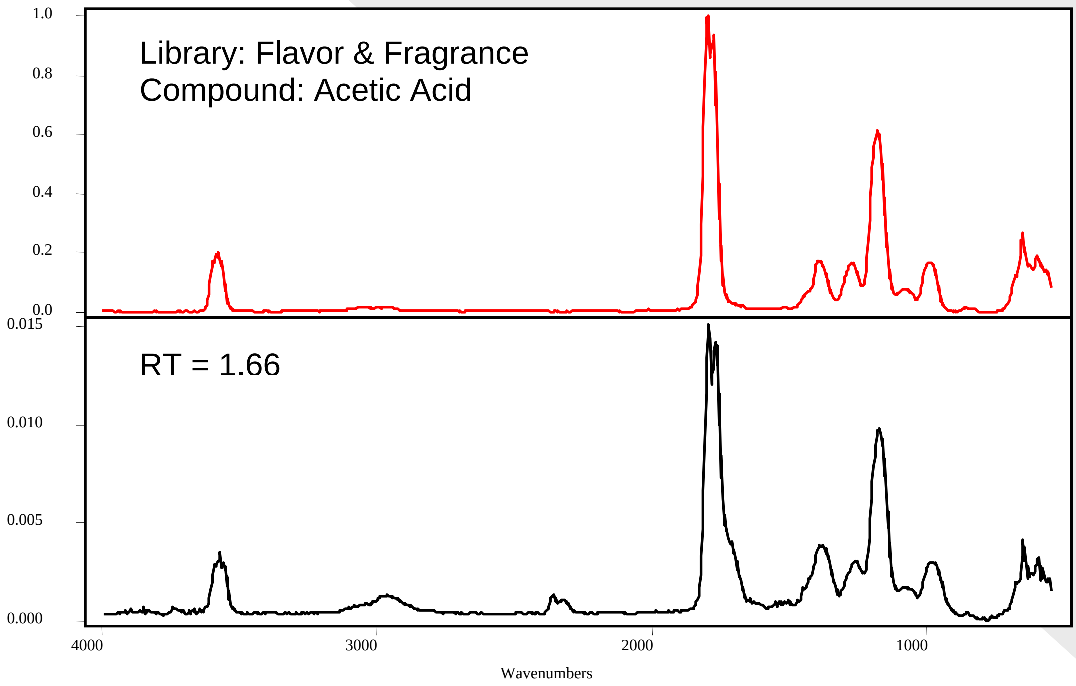
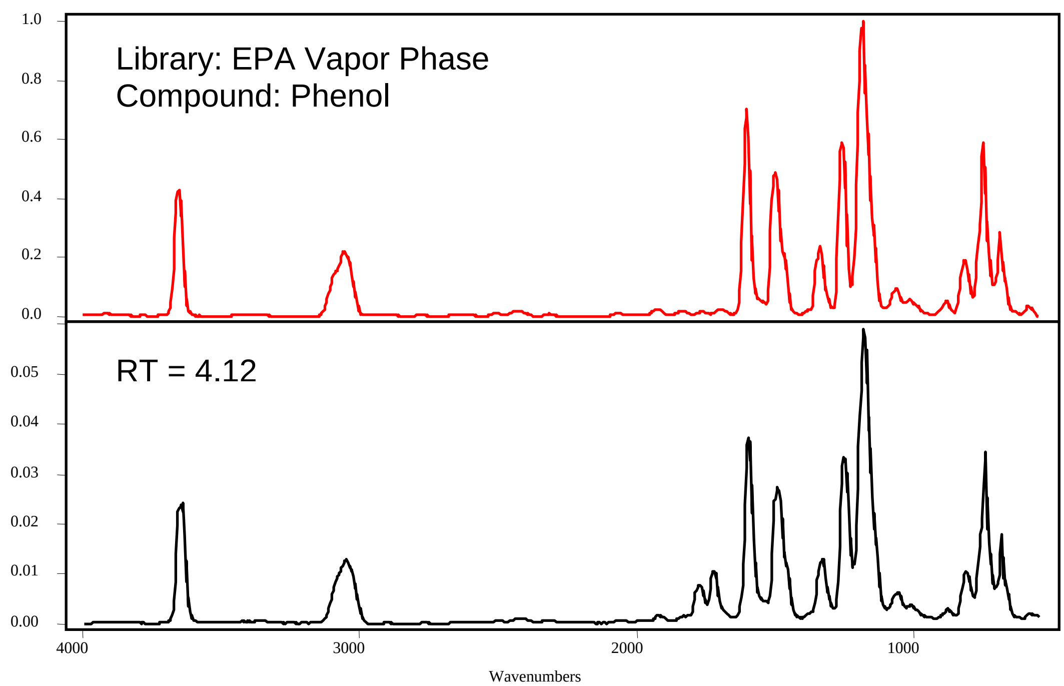
The IR spectra for carboxylic acids are clear, including the strong IR absorbance at 1750 wavenumbers for the carbonyl and the IR absorbance at 3600 wavenumbers for the –OH group. Here is acetic acid from these new fuel source samples:





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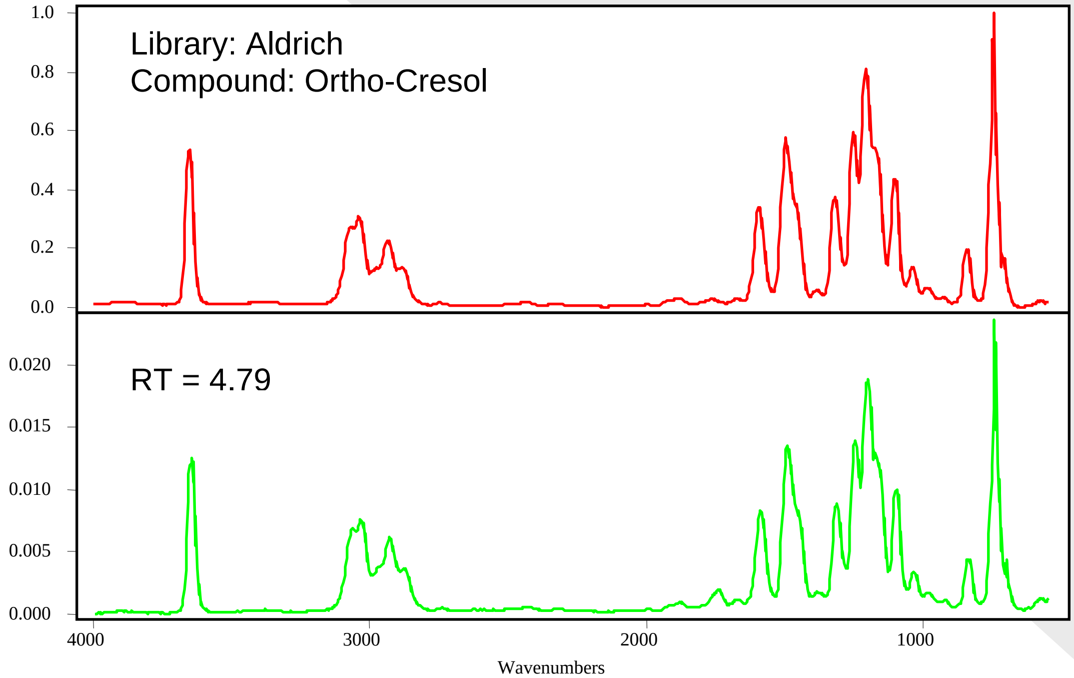
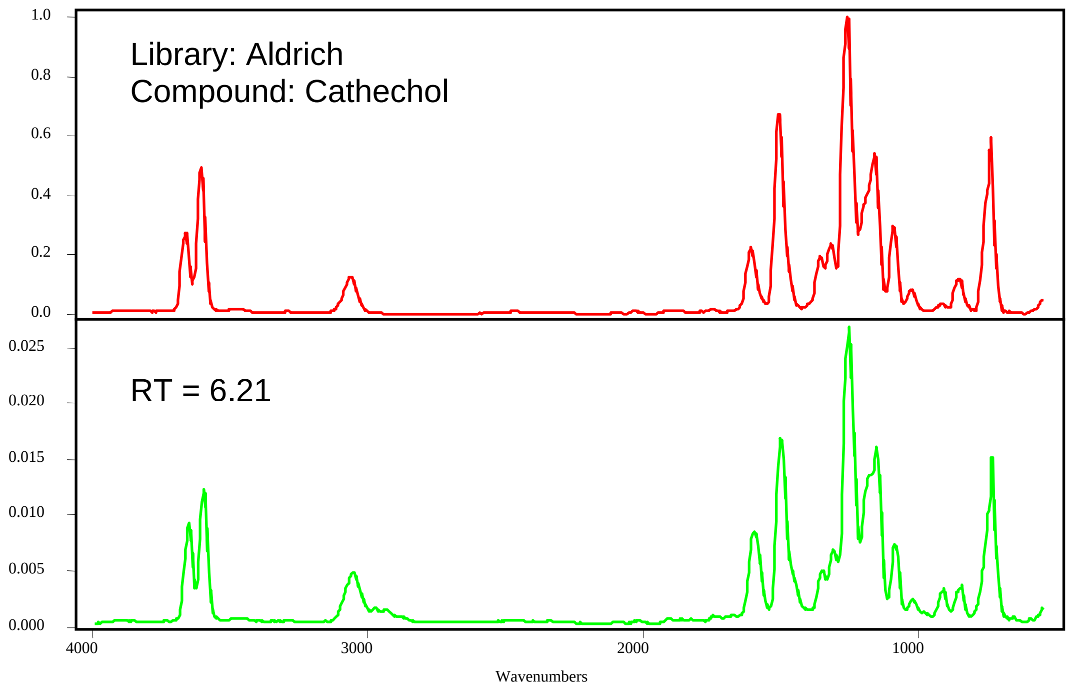
We can compare the spectra for phenol versus cresol with the additional absorbance features around 3000 wavenumbers. We can see the aromatic IR absorbance above 3000 wavenumbers in both spectra but only cresol has the aliphatic IR absorbance below 3000 wavenumbers:



We can compare the spectra for phenol versus catechol with a single versus a dual IR response in the 3600 wavenumber range.



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Conditions:

Gas Chromatograph: 40 to 260°C with split injection (5:1 and 0.5uL volume)
Column: Innowax 30m x .32 x 0.5
IRD: scan from 4000 to 550 wavenumbers
Optical Resolution: 8 wavenumber
Transfer Line Temperature: 280°C

Summary

It has been demonstrated that the GC-IRD system with the ASAP IRD 3 is very powerful tool for the analysis and characterization of new source fuel mixtures.

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