Fourier Transform Infrared Spectroscopy

Unknown Aerospace Contaminant Analysis

Macy Mullen, NE-L3 Purdue University Chemistry Mentor: Philip Howard

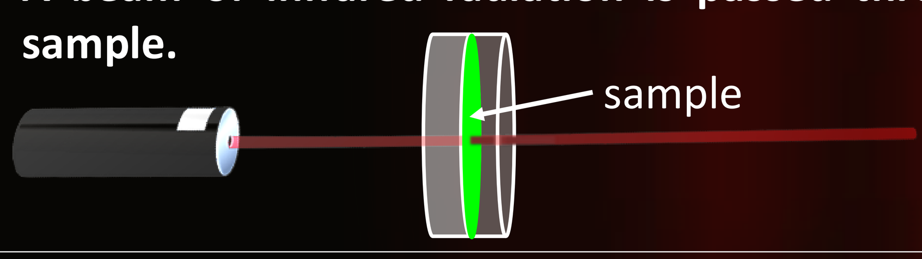
Introduction

KSC's Analytical Laboratories branch provides Center-wide chemical analysis support for the identification of contaminants found in payloads, flight hardware, clean rooms, and processing facilities for programs such as Orion, and almost every payload launched from KSC including commercial operations. This is a common but very serious issue and there are stringent health and safety restrictions pertaining to foreign object debris and contamination in aerospace systems due to their extremely sensitive nature and potential to interface with the astronauts aboard the ISS. When contamination is discovered, such as fibers, particles, residues, etc., it is sampled and then analyzed to determine its identity. Fourier Transform Infrared spectroscopy (FTIR) is a critical component of this analysis.

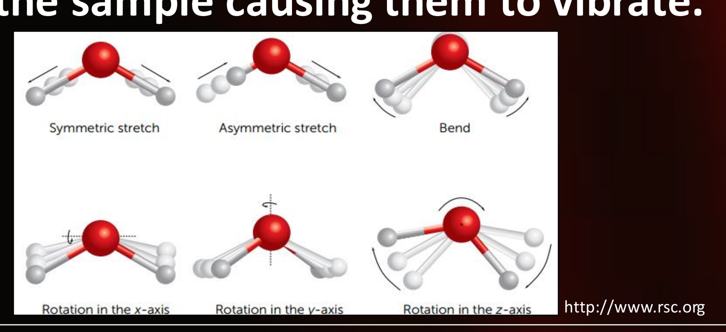
Background:

How does it work and what does it tell us?

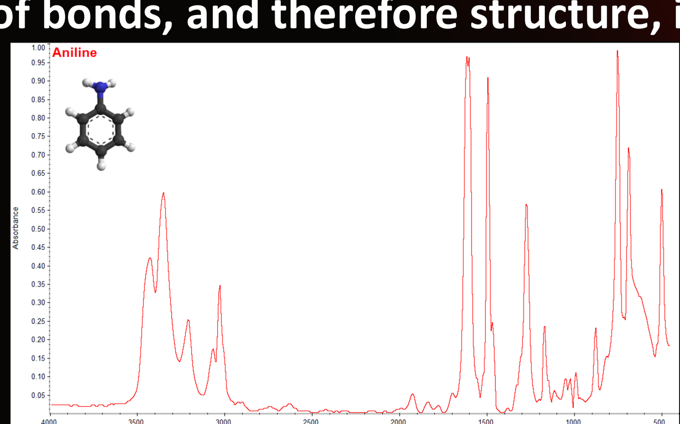
1. A beam of infrared radiation is passed through a sample.



2. The IR beam excites the chemical bonds in the molecules of the sample causing them to vibrate.



3. These vibrations absorb the infrared radiation at different wavelengths. Each molecule and material creates a unique absorption spectrum that indicates what sort of bonds, and therefore structure, it has.



These spectra indicate what sort of functional groups a material consists of. With that being known, scientists can use that data to extrapolate the structure and properties of unknown samples or newly synthesized compounds.

Spectral Interpretation: The Basics

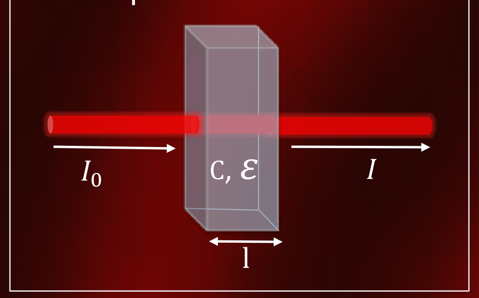
An IR spectrum displays the frequencies, measured in wavenumbers ($\tilde{\nu} = {}^{1}/_{\lambda} = \mathrm{cm}^{-1}$), that are being absorbed by the chemical bonds of a material. The percent absorbance, height of the peaks, follows the Beer's law (see Figure 1). What makes FTIR so advantageous is the wide range of materials capable of being analyzed using this technique, see below:

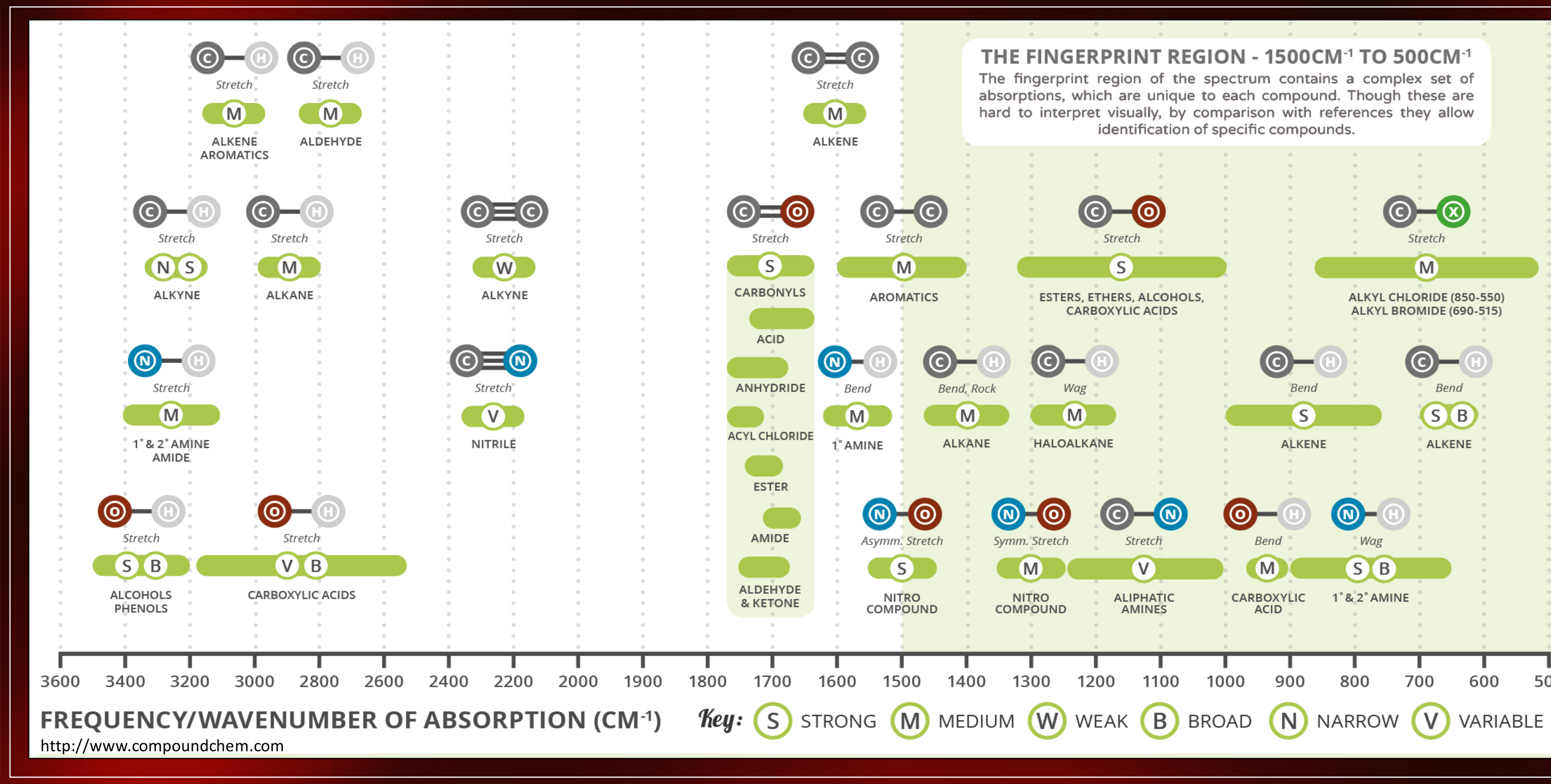
Figure 1. Beer's Law equation

Beer's Law

$$Absorbance = -\log\left(\frac{I}{I_0}\right) = \varepsilon\, l\, c$$

I_0 = light intensity initial
I = light intensity final
ε = sample molar absorptivity
l = sample path length
c = sample concentration

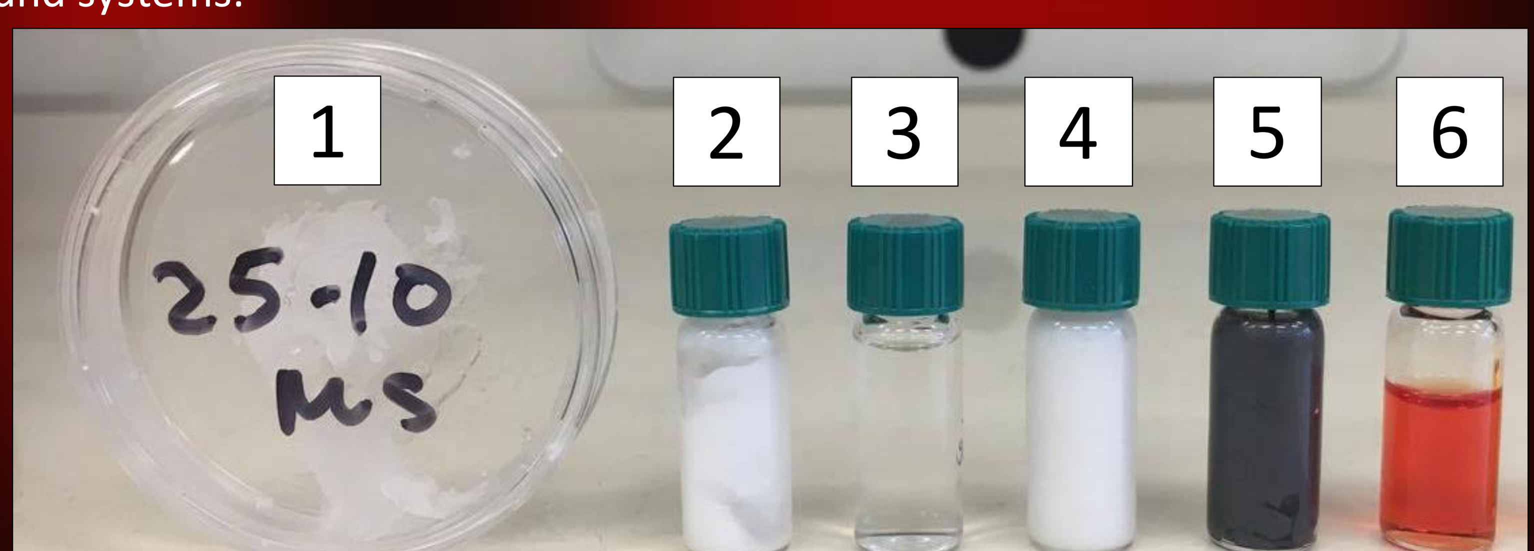




- Hydrocarbons
  - Aliphatic
  - Unsaturated
  - Aromatic
- Amines
  - Primary
  - Secondary
- Nitriles
- Alkyl Halides
- Ethers
- Alcohols
- Carbonyls
  - Ketones
  - Aldehydes
  - Esters
  - Amides
  - Carboxylic Acids
- Inorganics

Sample Analysis

Every year we receive hundreds of samples, all from different customers, facilities, projects, and systems. Samples range from simple greases and particulates to very complex mixtures of a number of different contaminants. The key to faster and more confident interpretation is to build your own internal database of spectra. This summer I conducted a number of different analyses, one being of aerospace greases, to familiarize myself with common greases, oils, and hydraulic fluids used in different flight and ground systems.



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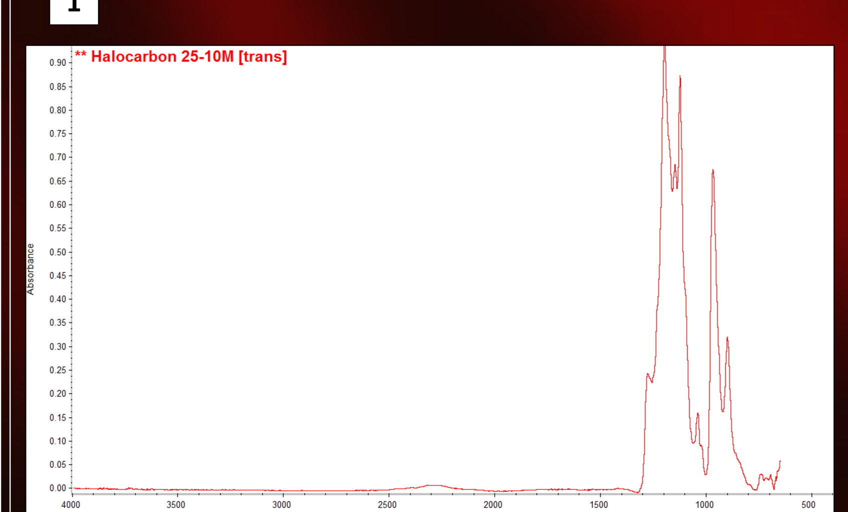

Halocarbon®25-10M is polychlorotrifluoroethylene (PCTFE). This grease is commonly found in oxygen systems, from the Apollo ground systems to the ISS. The halogenated nature (Cl and F) of this grease makes it compatible with systems handling oxidizers, unlike hydrocarbon greases.

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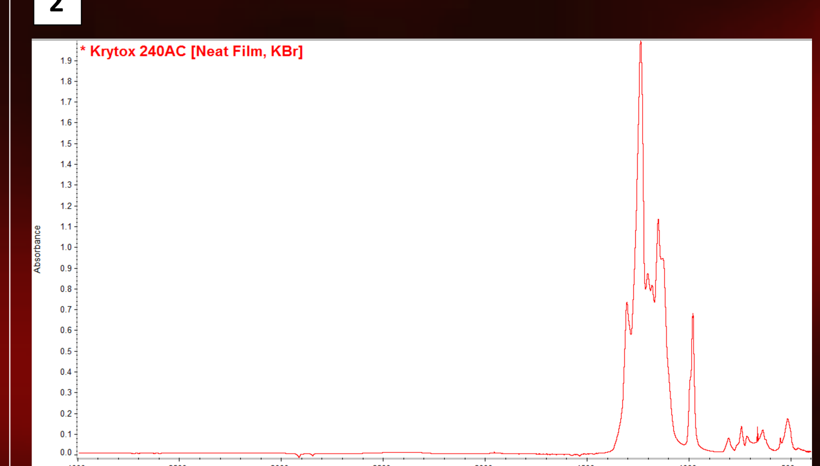

Krytox®240AC is a common aerospace lubricant used in numerous vehicles including the space shuttle. It is a perfluoropolyether (PFPE) based grease thickened with polytetrafluoroethylene (PTFE) capable of withstanding extreme conditions, is chemically inert, and one of the original greases to meet military specifications for fuel and oxidizer resistance.

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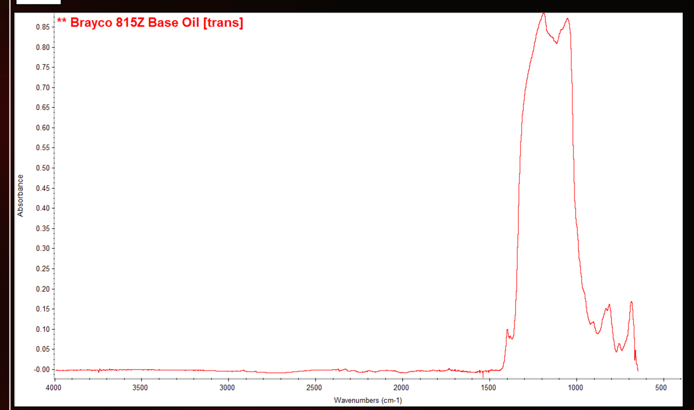

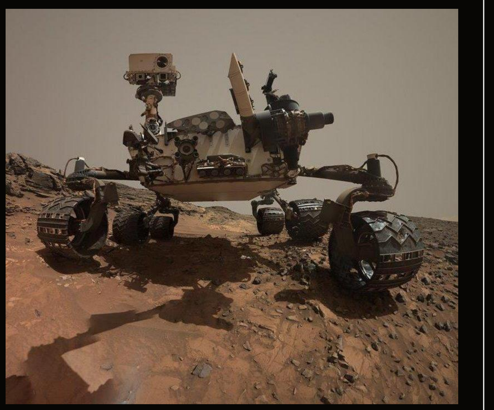

Brayco®815Z is a PFPE base oil for Braycote® 600EF and 602EF. It is compatible with aerospace fuels and ,due to its chemical inertness, is unaltered by UV and cosmic radiation, high vacuums, and low temperatures. One of the more famous places it can be found is the gearboxes on the Mars Curiosity rover.

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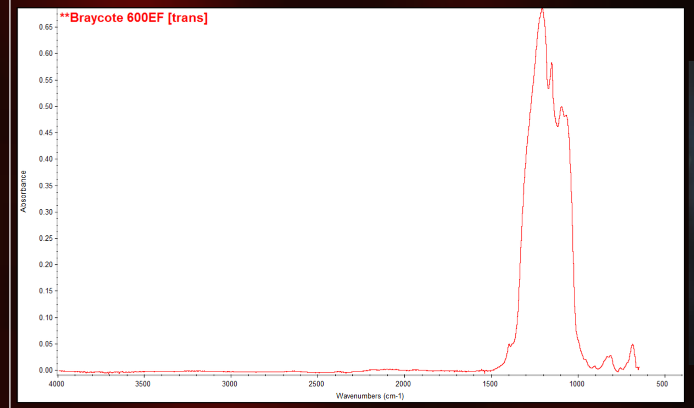

Braycote®600EF is Brayco®815Z thickened with polytetrafluoroethylene (PTFE). This grease acts as both a lubricant and corrosion inhibitor for the ISS.

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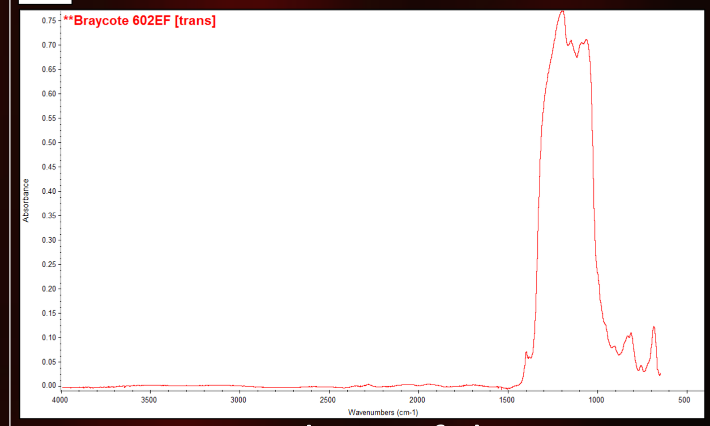

Braycote®602EF is Braycote®600EF with molybdenum disulfide (MoS_2). This additive is indicative of extreme pressure environments where if the base oil were squeezed out of the system, the MoS_2 particles would act as a dry lubricant.

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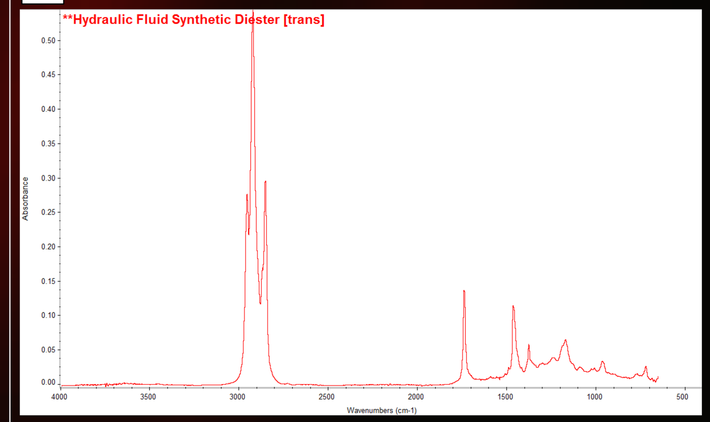

This synthetic diester hydraulic fluid is found all over center in a variety of different programs, such as the Delta II launch vehicle. Even though this vehicle is now retired, knowing which facilities house certain materials helps to determine whether a contaminant is entirely foreign or migrated from somewhere else on the system.

References

Howard, P. M. Training. NASA Kennedy Space Center.
Mullen, M. (2016). *Pathways intern report*. John F. Kennedy Space Center, NASA.

Acknowledgements

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