

About OMICS Group

OMICS Group International is an amalgamation of Open Access publications and worldwide international science conferences and events. Established in the year 2007 with the sole aim of making the information on Sciences and technology 'Open Access', OMICS Group publishes 400 online open access scholarly journals in all aspects of Science, Engineering, Management and Technology journals. OMICS Group has been instrumental in taking the knowledge on Science & technology to the doorsteps of ordinary men and women. Research Scholars, Students, Libraries, Educational Institutions, Research centers and the industry are main stakeholders that benefitted greatly from this knowledge dissemination. OMICS Group also organizes 300 International conferences annually across the globe, where knowledge transfer takes place through debates, round table discussions, poster presentations, workshops, symposia and exhibitions.

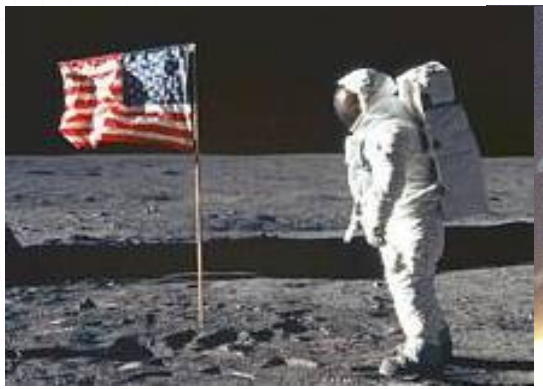
About OMICS Group Conferences

OMICS Group International is a pioneer and leading science event organizer, which publishes around 400 open access journals and conducts over 300 Medical, Clinical, Engineering, Life Sciences, Phrama scientific conferences all over the globe annually with the support of more than 1000 scientific associations and 30,000 editorial board members and 3.5 million followers to its credit.

OMICS Group has organized 500 conferences, workshops and national symposiums across the major cities including San Francisco, Las Vegas, San Antonio, Omaha, Orlando, Raleigh, Santa Clara, Chicago, Philadelphia, Baltimore, United Kingdom, Valencia, Dubai, Beijing, Hyderabad, Bengaluru and Mumbai.

Precise Heat Control. Essential Materials Characterization Techniques Everyone Needs to Solve Real Problems

Rodrigo Devivar, Ph.D.
Jacobs Technology / NASA-Johnson Space Center
ES4 Materials Analysis Laboratory
Houston, Texas





Controlling Heat in Aerospace



Picture of Space Shuttle During Atmospheric Re-entry taken from ISS







Analytical Chemistry Laboratory Equipment







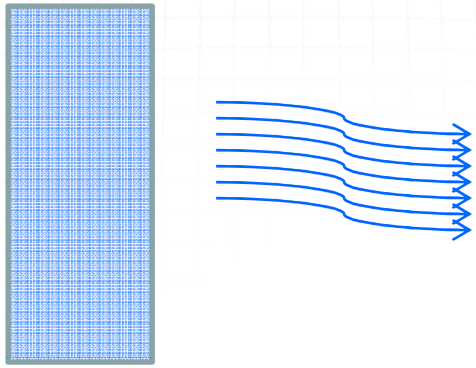
Key Laboratory Equipment

- Optical Instrumentation
  - UV-Vis, Fluorimeter, Solar Reflectance, Infrared Emittance, Raman
- Thermal Analysis Instrumentation
  - DSC, DMA, TGA, TMA, LFA, Rheometer
- Chemical Analysis Instrumentation
  - FT-IR, Ion trap GC-MS, Py-GC-MS, TGA-MS, TGA-IR



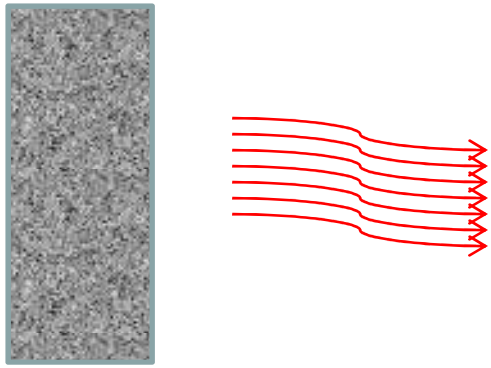
Optical Vs. Thermal Techniques

Light



Reflectance
Emittance
Absorbance/Transmission
Fluorescence
UV-Vis Absorbance
FT-IR Analysis
Raman Analysis

Heat

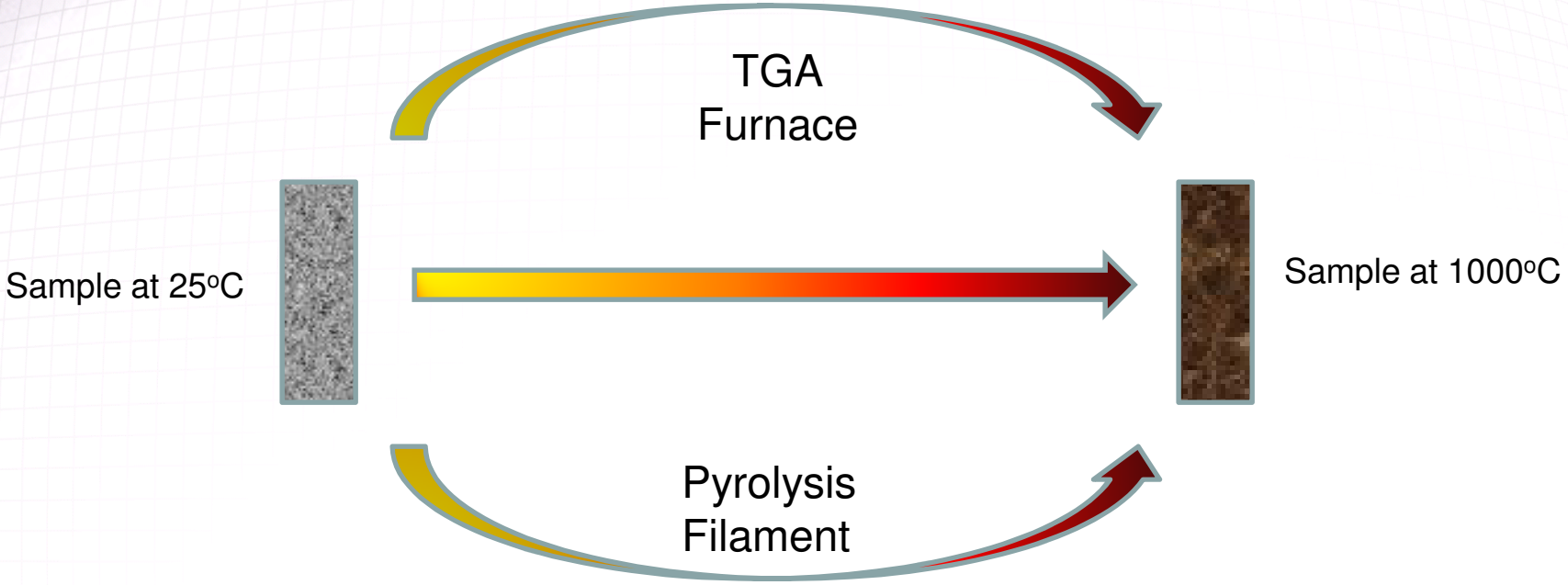


Material Curing
Thermal Transition-Tg
Melting Point/ Boiling Point
Residual Solvent
Identification of additives
Material Decomposition
Elimination of labile functional groups
Identification of Material Components
Identification of Inorganic Components

Controlling Heat Exposure

Thermal Analysis

Slow: minutes to hours



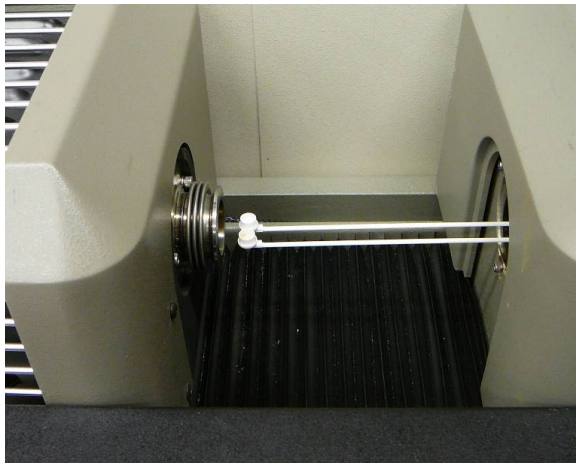
Fast: microseconds to seconds

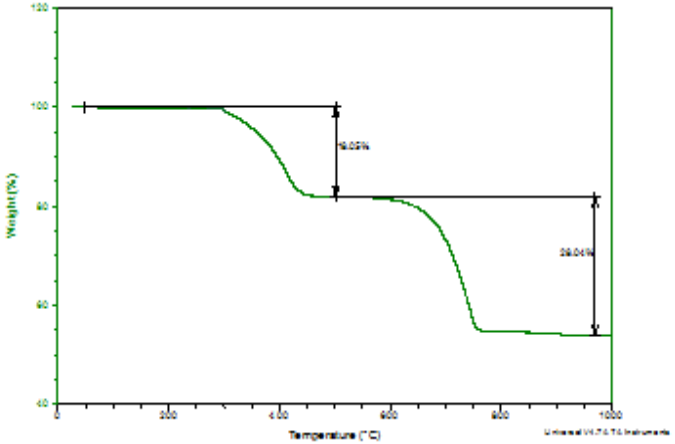
Thermochemical Analysis

Thermogravimetric Analysis (TGA)

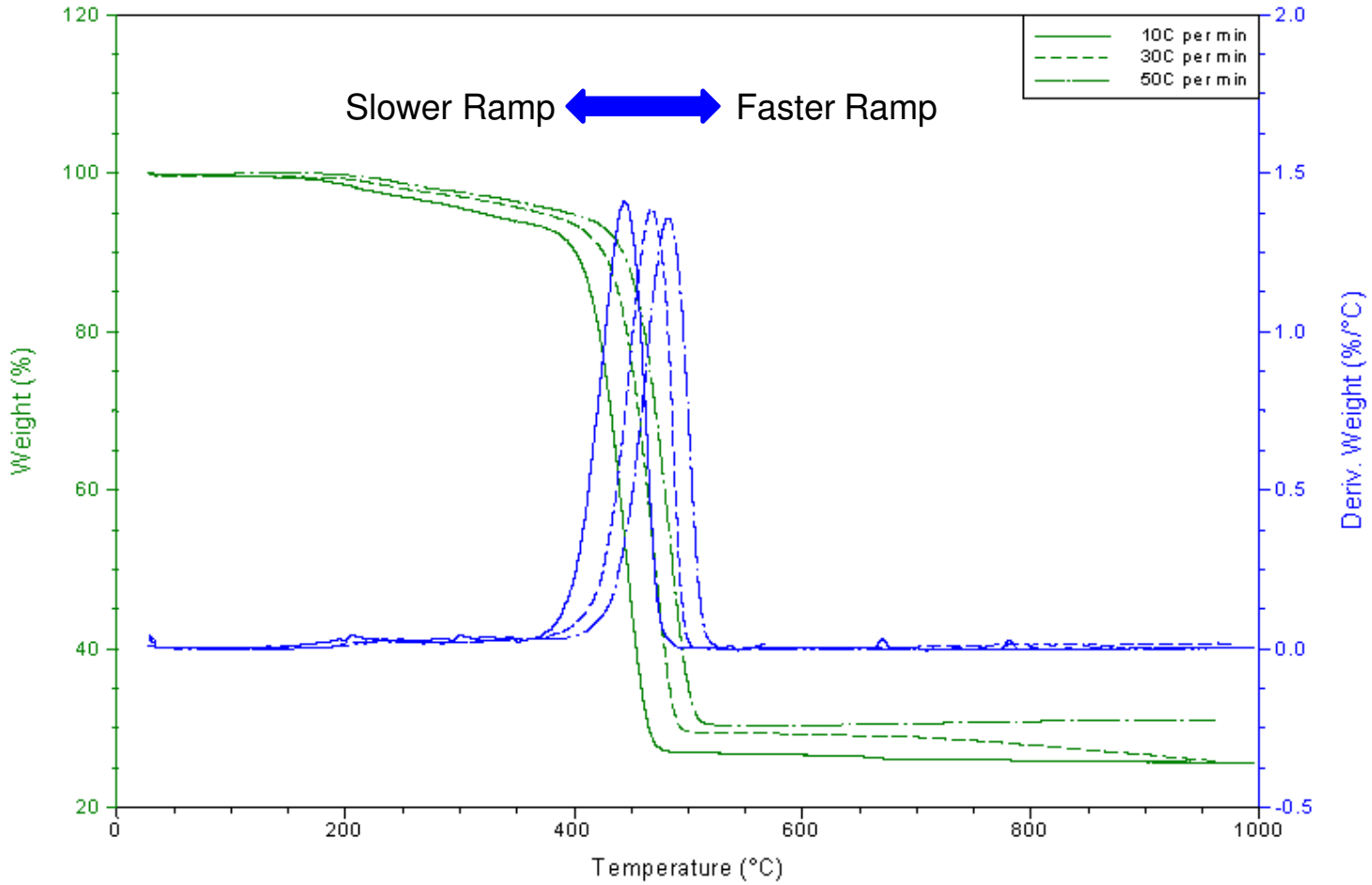
- A TGA instrument consists of an analytical balance and a furnace.
- A small sample of material is heated and its change in mass is measured as a function of temperature.
- Experiments can be conducted under inert or oxidizing atmospheres.
- Information gained from TGA includes:
  - Thermal stability for conducting additional thermal analysis
  - Identification of the number of components in the sample if the decomposition temperatures are different
  - Residual mass for assessing the extent of inorganic additives







The Influence of Temperature Ramp Rates

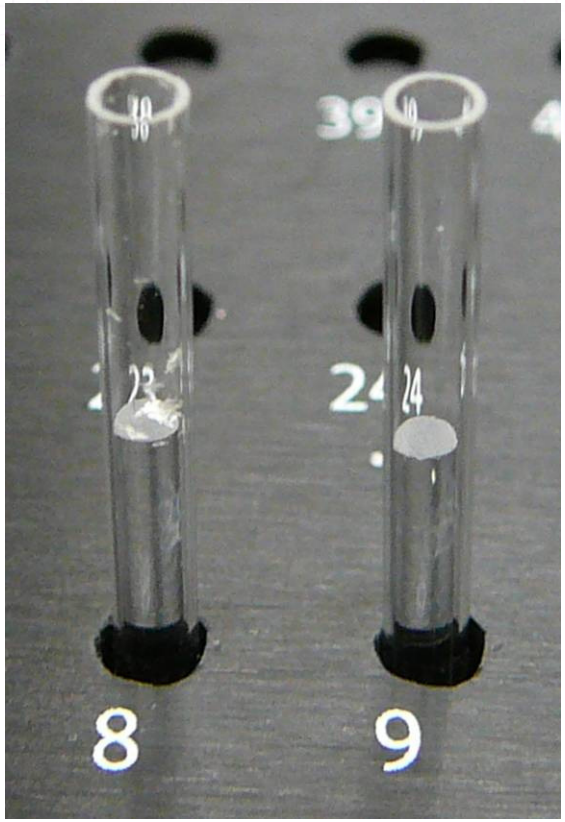


Pyrolysis for GC-MS of Solids

- Sample size is relatively small:

  50 to 200 µg is sufficient for solids
  50 to 200 nL is sufficient for liquids

- Sample preparation is easy: Place sample inside 1.5 inch quartz tube containing filler tube and plug with glass wool.
- Samples can be solids, gels, viscous liquids, greases, crystalline, emulsions, foams, fabrics
- Pyrolysis temperatures are almost instantaneous
- Sample components can be quantified with the use of software





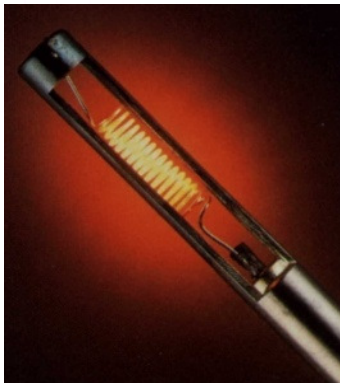
Pyrolysis is the thermal degradation of any substance through the fast application of heat.

Pyrolyzers: Filament Versus Furnace

CDS Platinum Filament

- Heating Rate: ~20,000°C per sec
- Max Temperature: 1400°C
- Cooling Rate: > 1000°C per sec

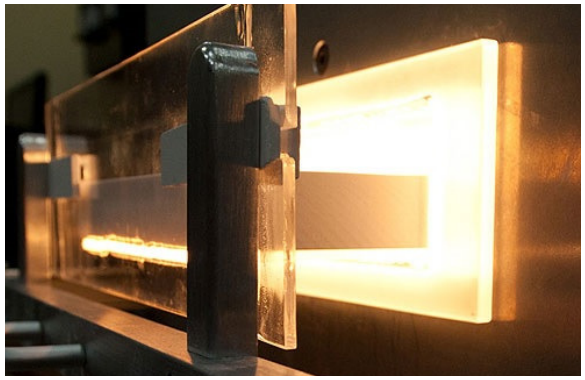
- Fast Heating, Fast Cooling



Microfurnace

- Heating Rate: ~50°C per min
- Max Temperature: 800°C
- Cooling Rate: 25°C per min

- Slow to Heat, Slow to Cool



Flash Pyrolysis of Polymers

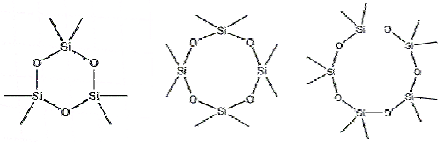
Mechanism of Pyrolytic Degradation

Random Scission

Unzipping

Side Group Elimination

Silicone Scission



Polyethylene: $-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-$

TIC; PTFE 750 3min.xms; Filtered

PTFE : $-CF_2-CF_2-CF_2-CF_2-CF_2-CF_2-CF_2-CF_2-CF_2-$

TIC; PVC test SP2 750.xms; Filtered

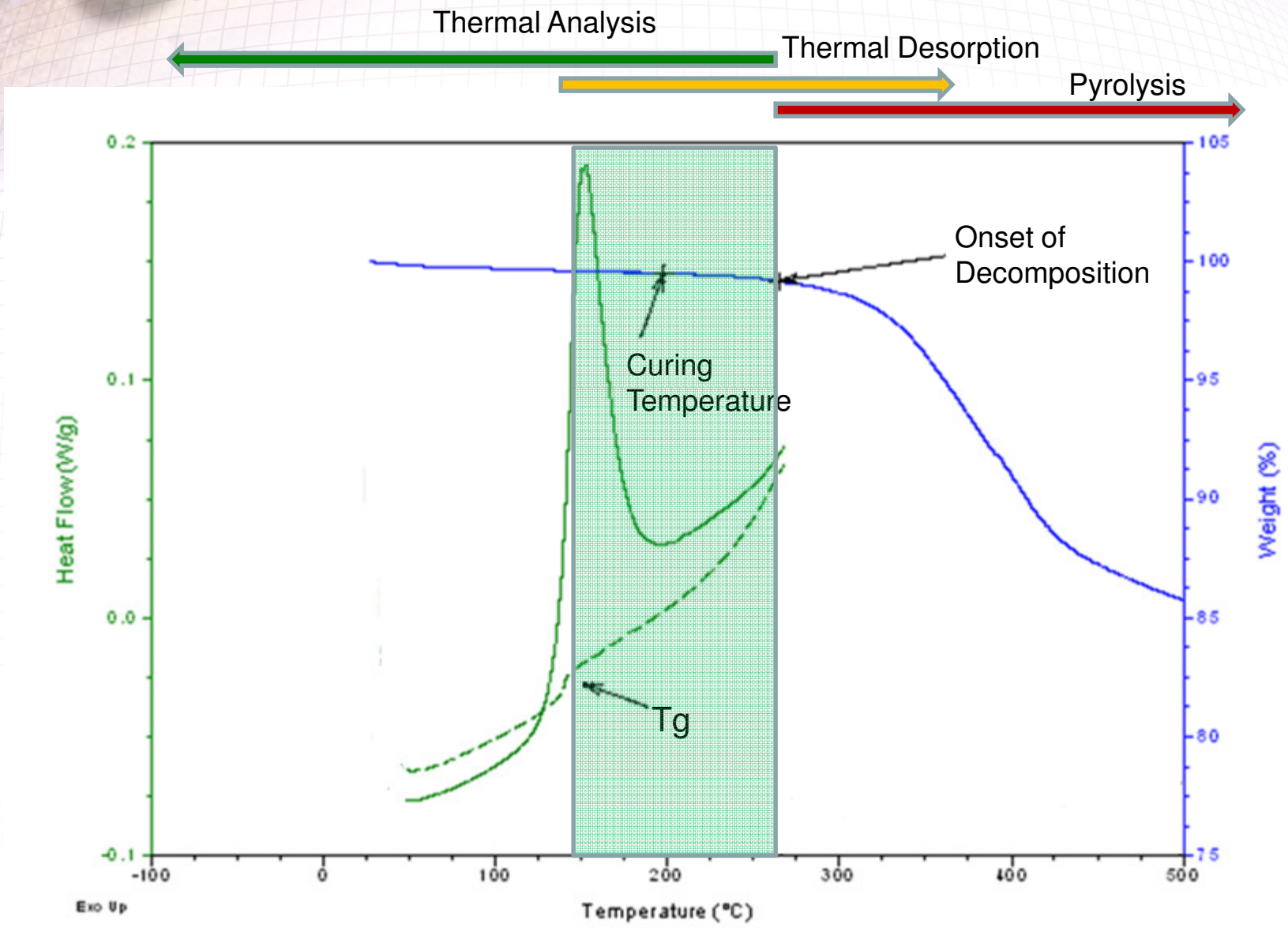
PVC: $-CH_2-CH(Cl)-CH_2-CH(Cl)-CH_2-CH(Cl)-CH_2-CH(Cl)-CH_2-CH(Cl)-$

TIC; PDMS std.XMS; Filtered

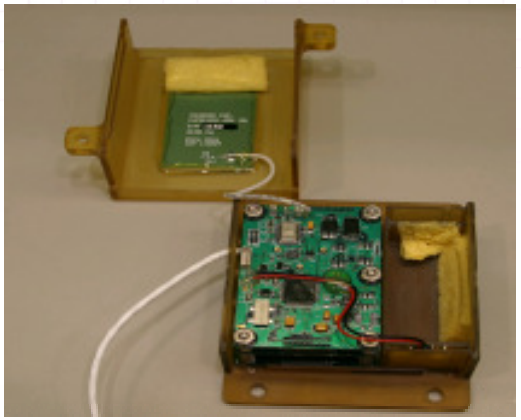
Silicones : $-O-Si(CH_3)_2-O-Si(CH_3)_2-O-Si(CH_3)_2-O-Si(CH_3)_2-$

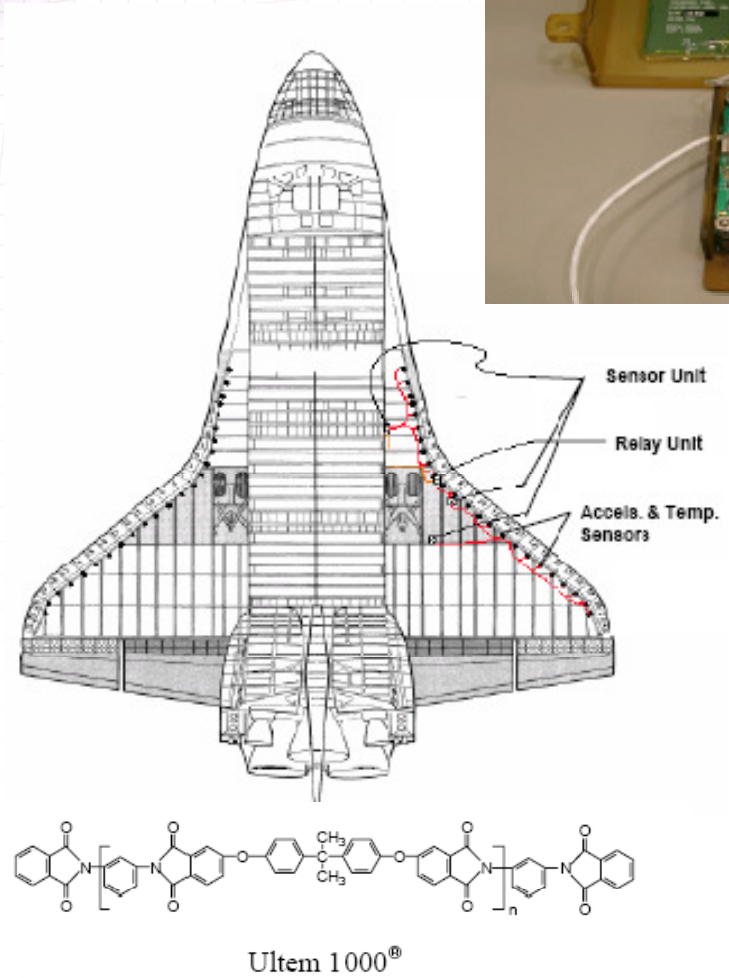
5.0 7.5 10.0 12.5 15.0

Thermal Analysis of Composite



Flash Pyrolysis-GC-MS of Ultem 1000



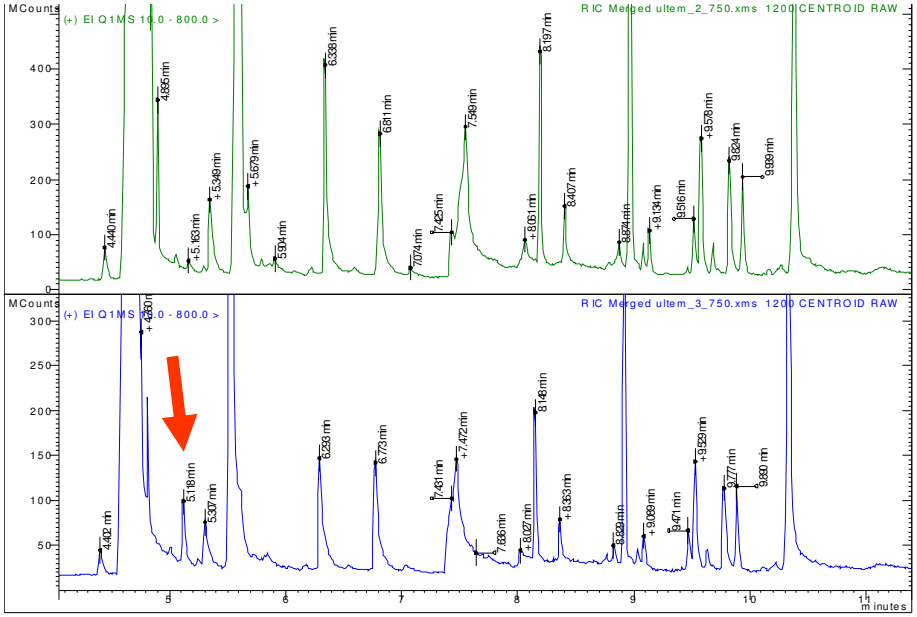


Ultem 1000®

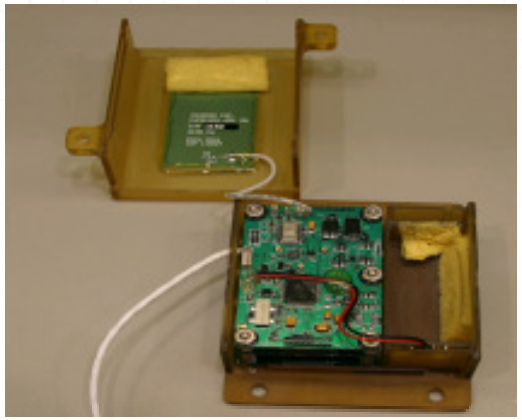
Relay sensor boxes along the shuttle's wing leading edge were composed of Ultem 1000.

One lot used to make these relay sensor boxes had failed

Various manufacture lots of sensor boxes were analyzed by Py-GC-MS and an extra peak was noted in one of those lots. The extra peak was due to dichlorobenzene, a solvent used during manufacture of Ultem 1000.



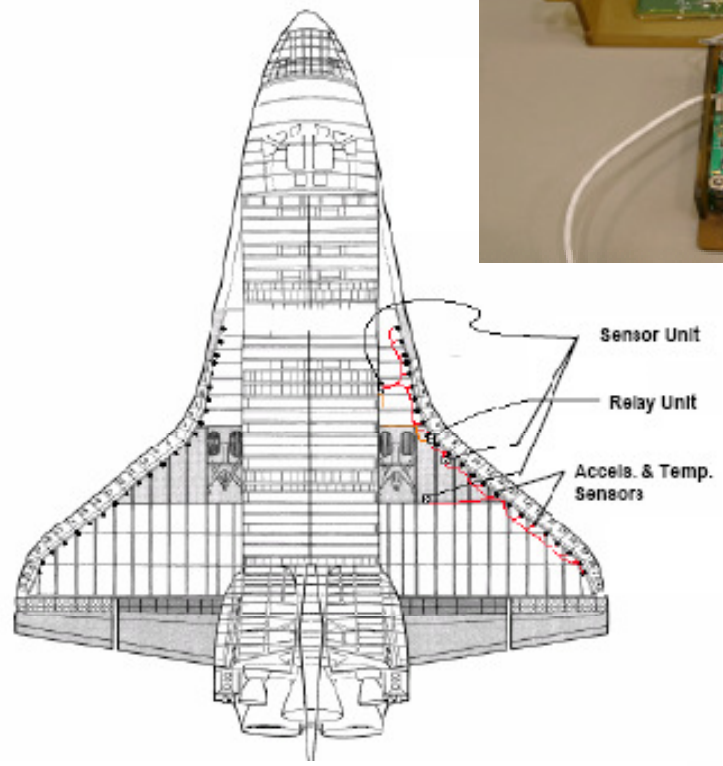
Thermal Analysis of Ultem 1000



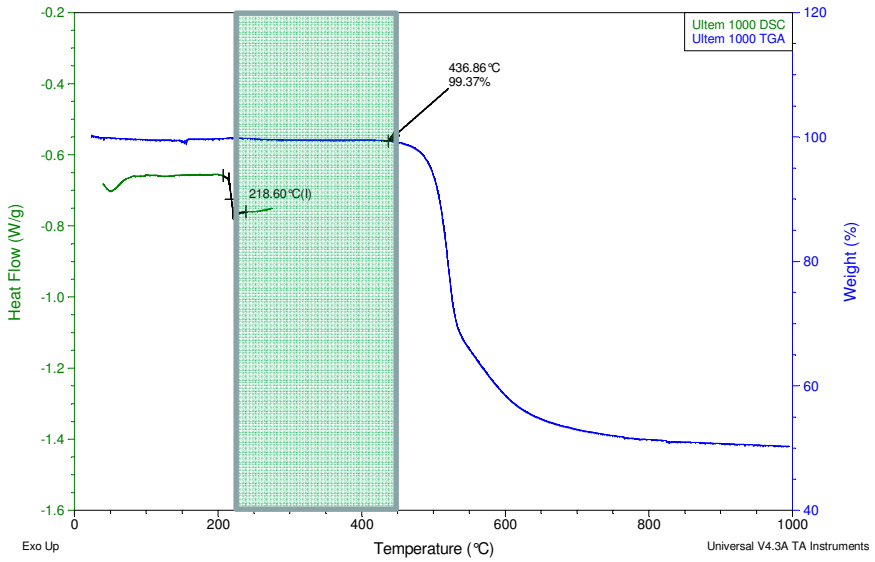
Key thermal values can be measured or obtained from product specification information.

Ultem 1000 Tg: 218°C

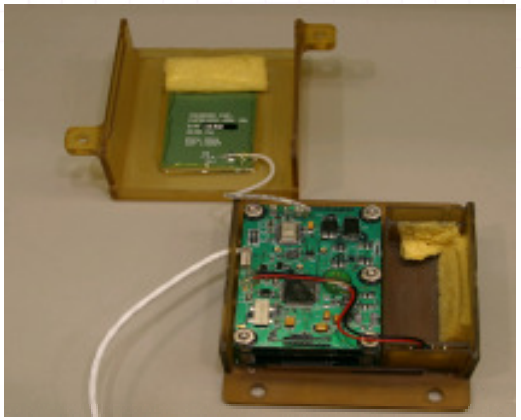
Onset of Decomposition: 437°C



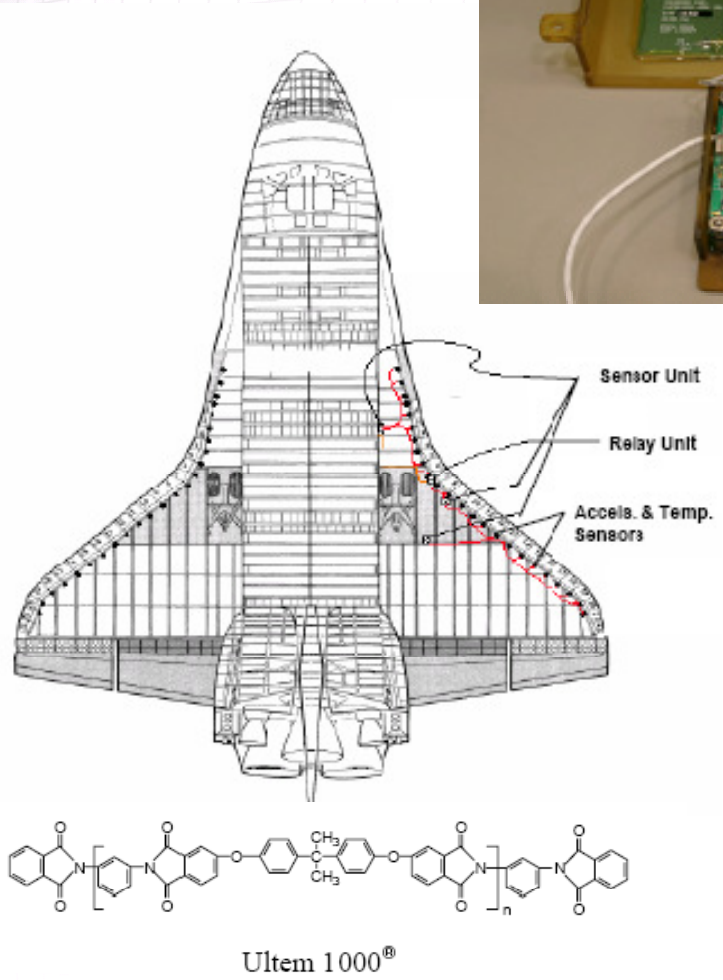
Ultem 1000®



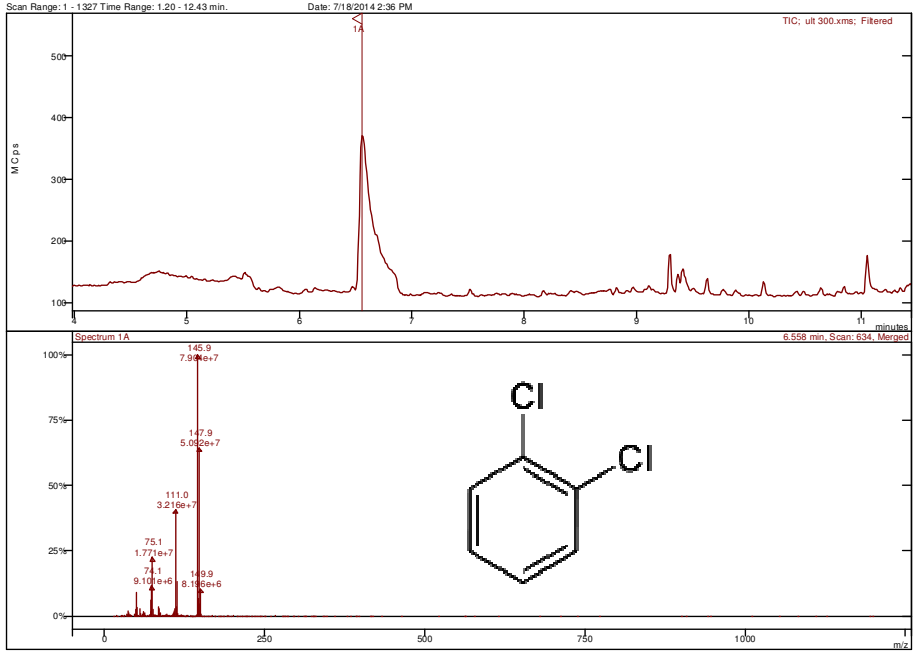
Thermal Extraction of Ultem 1000



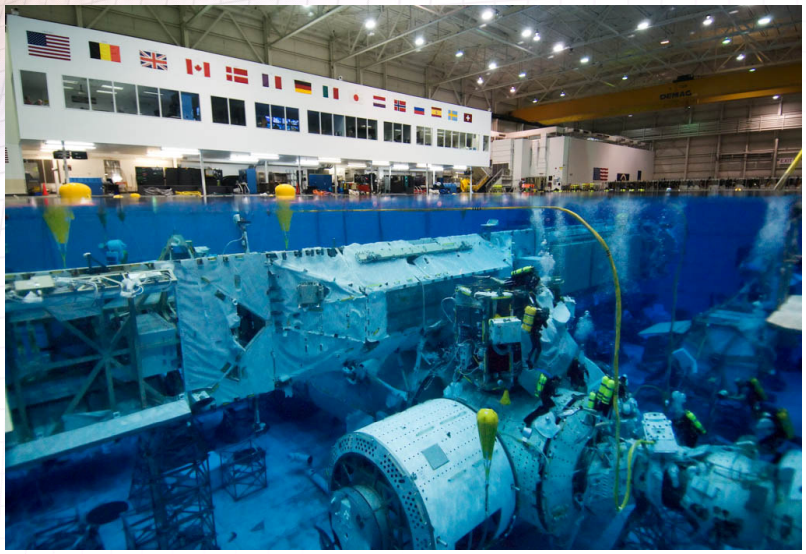
Thermal extraction temperature: 400°C



Ultem 1000®



Neutral Buoyancy Training Facility



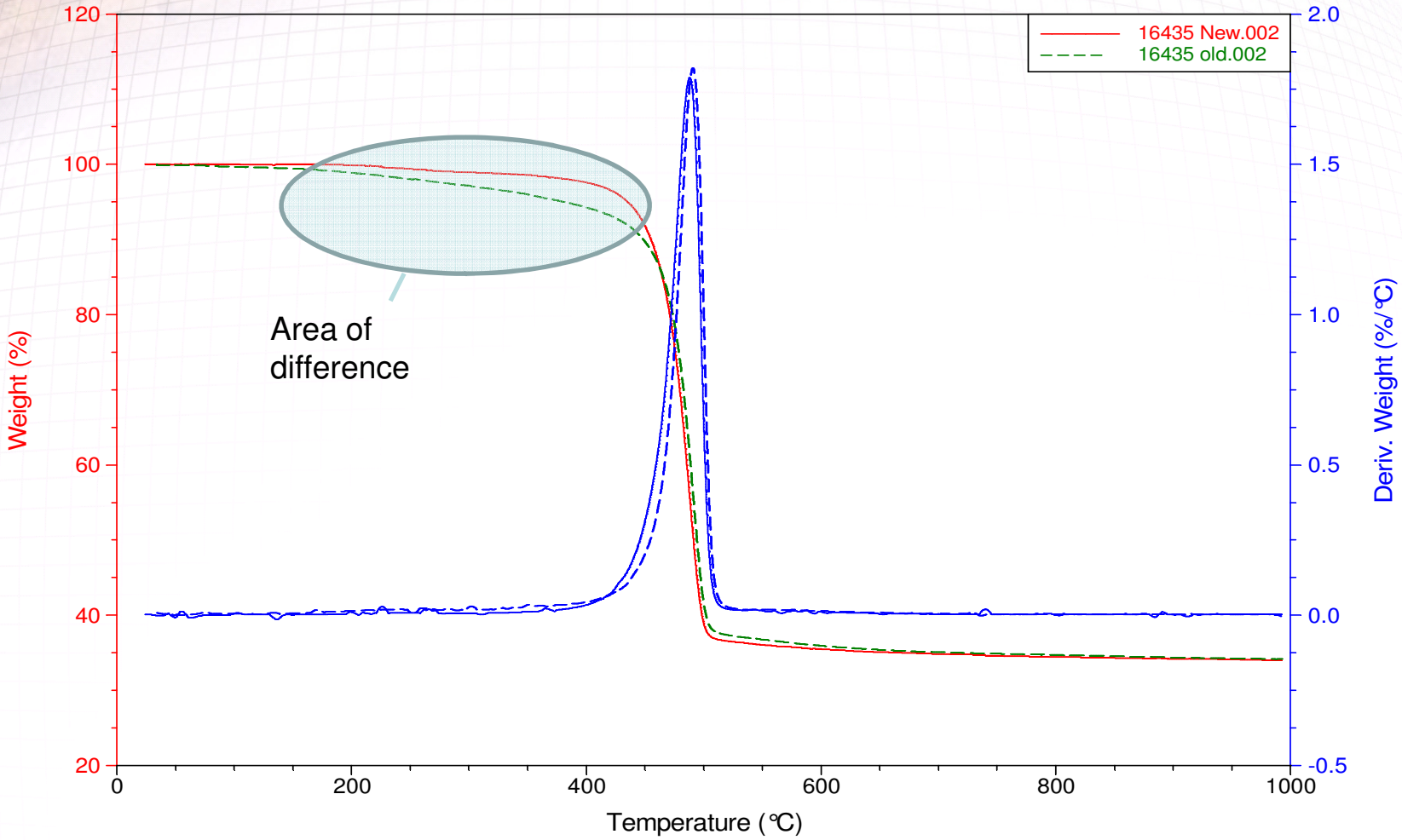


The NBL tank is 202 feet (62 m) long, 102 feet (31 m) wide, and 40 feet 6 inches (12.34 m) deep, and contains 6.2 million gallons (23.5 million litres) of water.

The facility is essential for astronaut EVA training prior to a mission.

On one occasion, a Viton gasket from the Canadarm hydraulics swelled and failed. We implemented a few thermal techniques to reveal the cause of the failure.

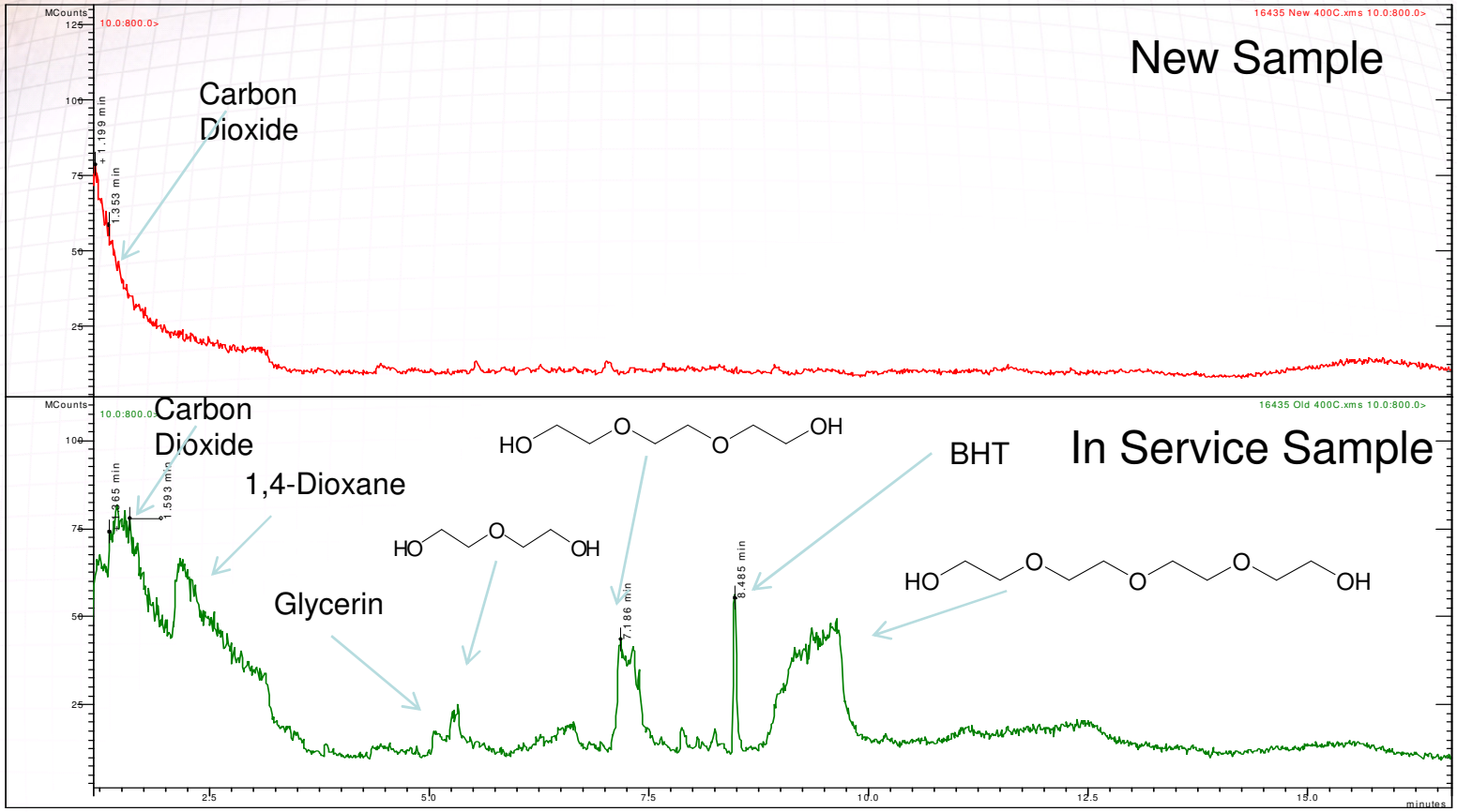
TGA Comparison of Gaskets



Under conditions of increasing temperature, the only difference between the two Viton Gaskets was found below 400°C, where the old sample lost a larger percentage of its mass compared to the new sample.

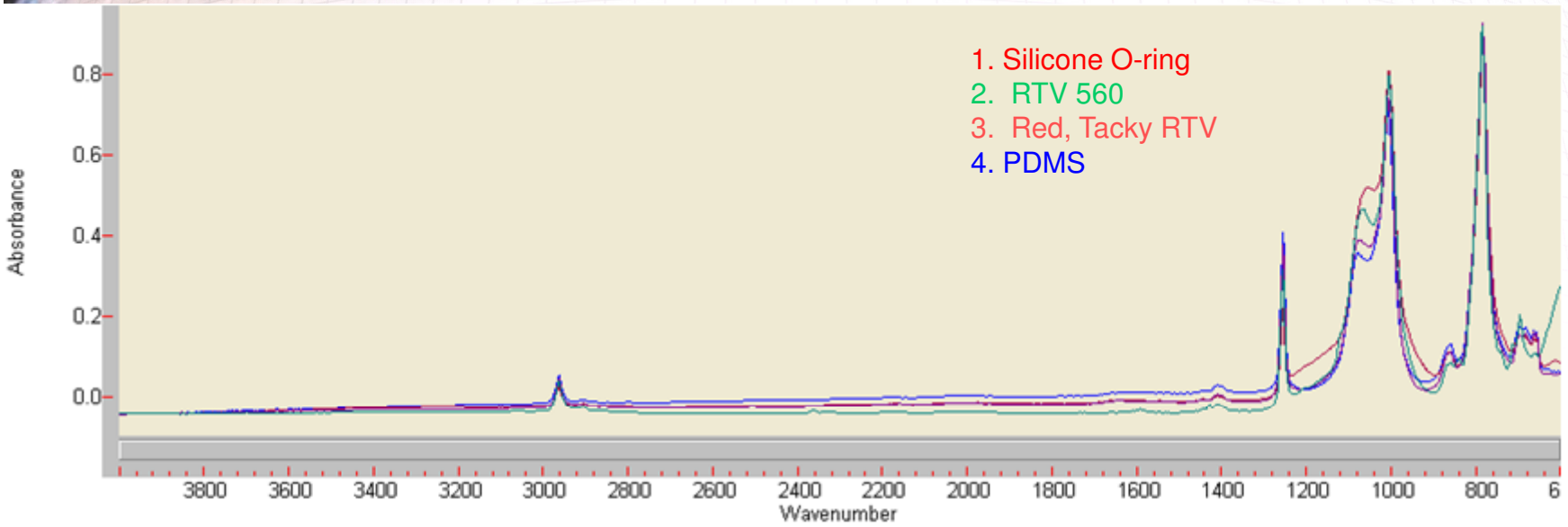
Thermal Extraction of Samples

Chromatogram Plots

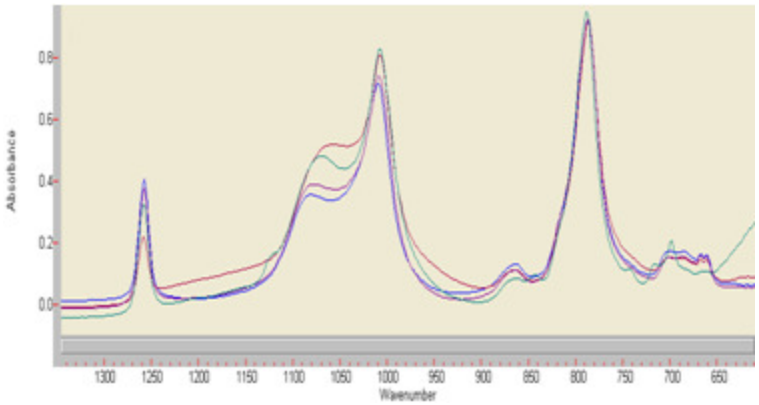


Thermal extraction of the two samples was performed to account for the difference observed in the TGA experiments at temperatures below 400°C. Such an experiment indicated the Old sample contained various fragments that are attributed to polyethylene oxide. Other substances found included Glycerin and Butylated hydroxy toluene (BHT).

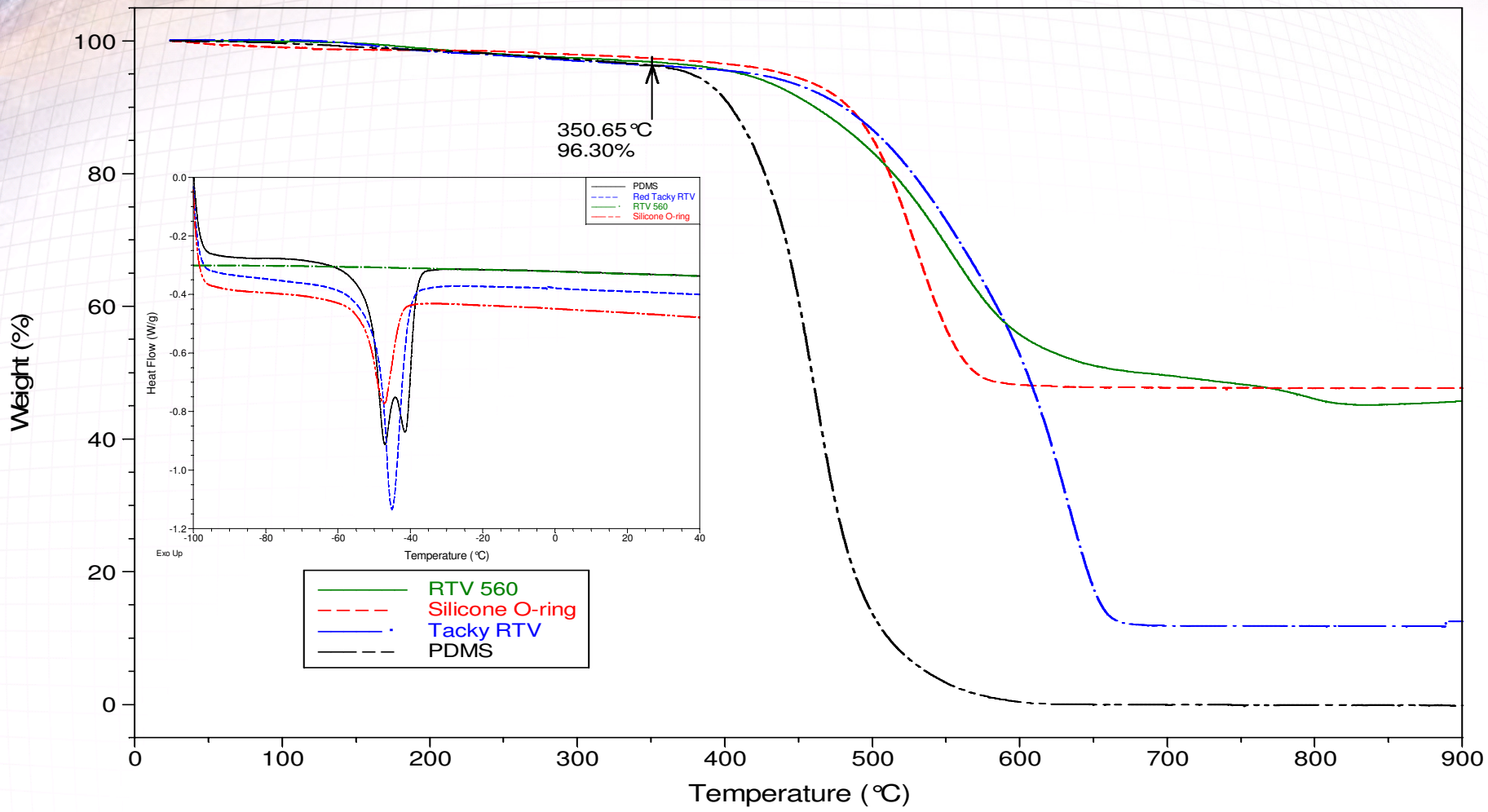
FT-IR Analysis of Silicone Materials



FT-IR is a non-destructive technique that is very diagnostic. However, if infrared light cannot penetrate the sample, any signal obtained through reflectance is only valid for the external surface of a sample.

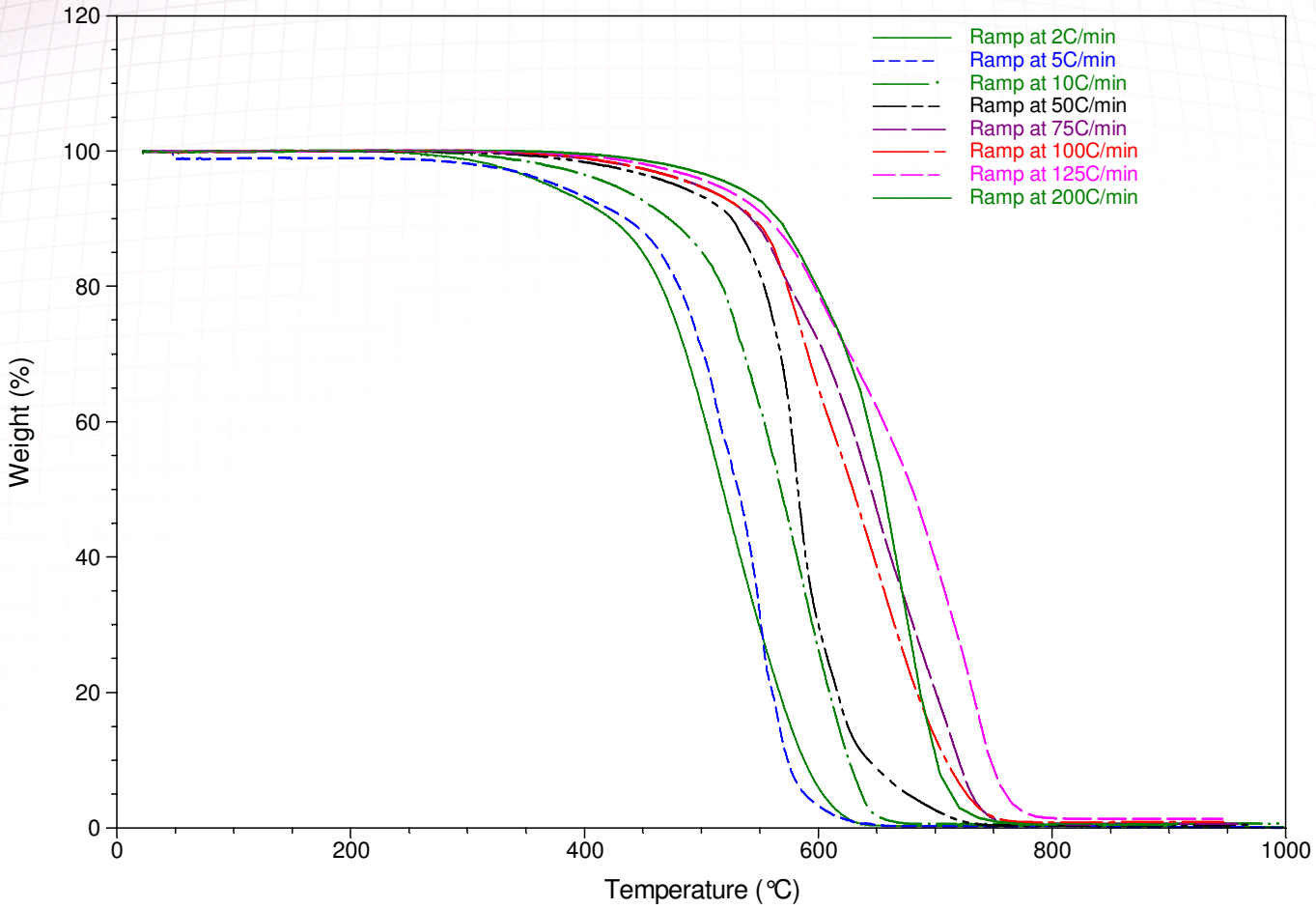


Thermal Analysis of Silicone Materials



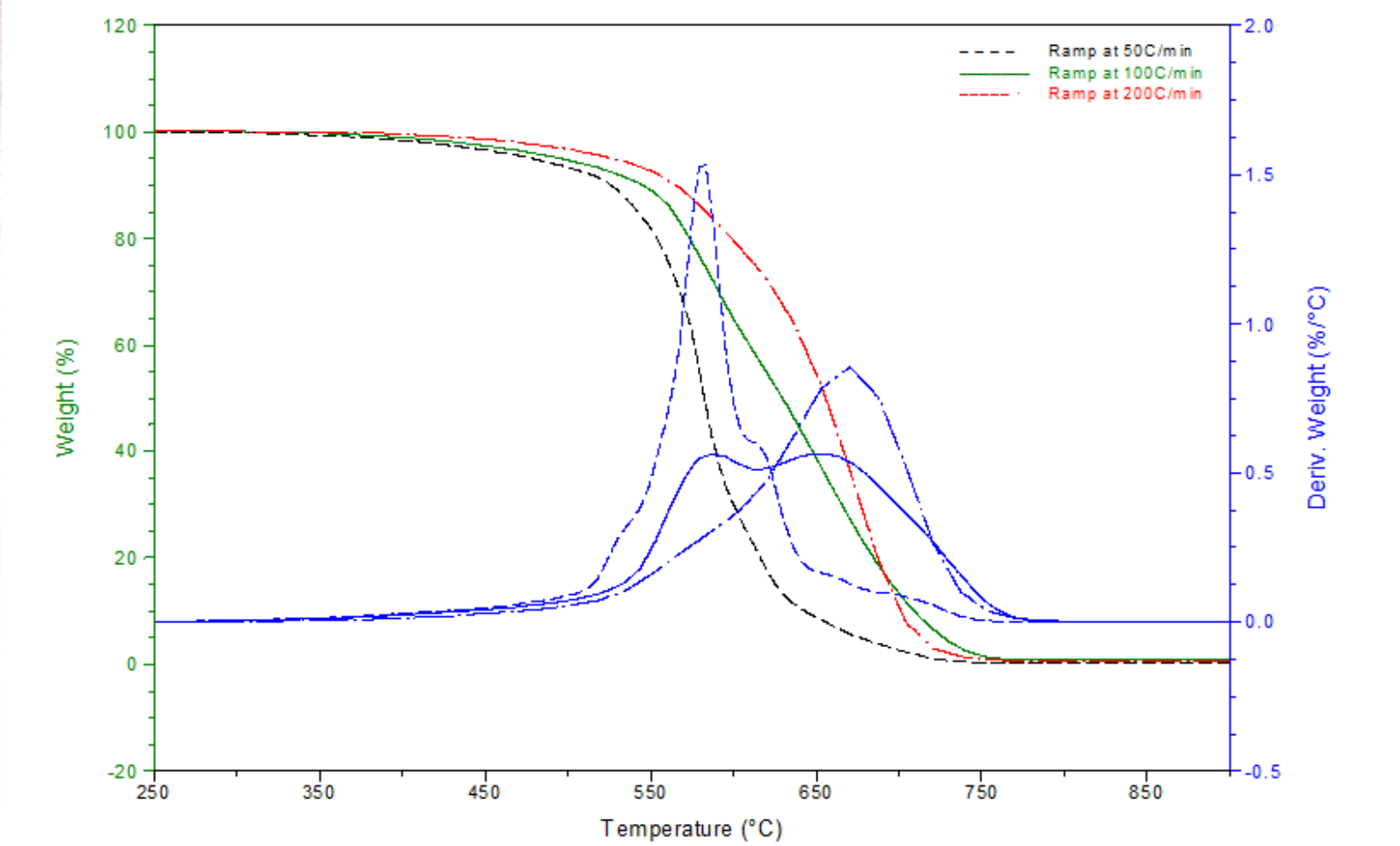
The Silicone samples that were nearly identical by FT-IR displayed very different properties by thermal analysis.

TGA Analysis of Silicone Oil



The thermal profile of silicone oil at different ramp rates indicates the complexity of thermal analysis

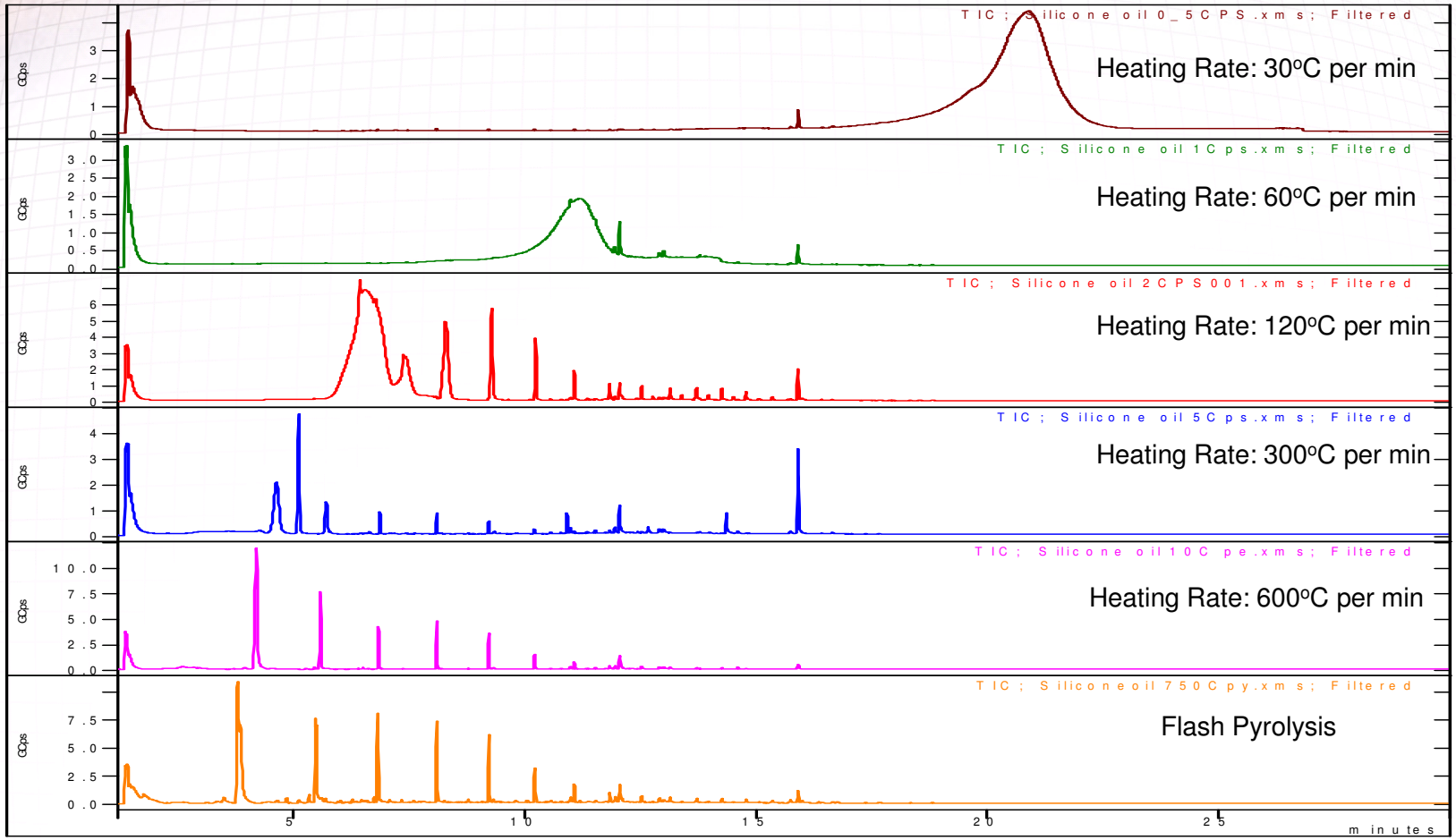
High Temperature Ramp Rates Increase Molecular Fragmentation



At high temperature ramp rates, silicone oil undergoes two types of processes detected by TGA analysis, evaporation through boiling and molecular fragmentation.

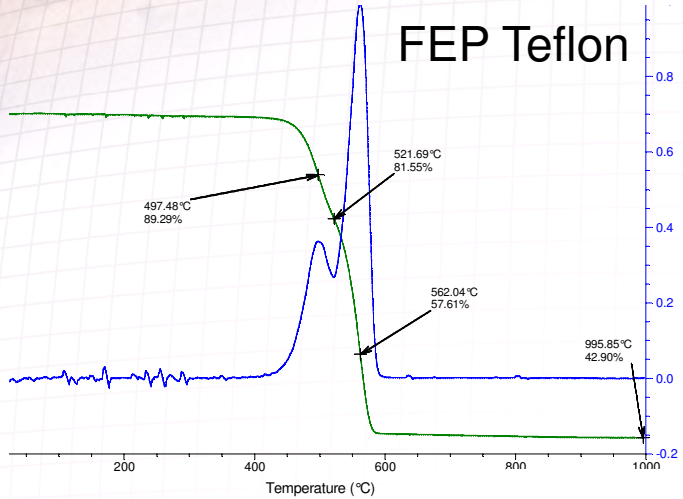
Pyrolytic Analysis of Silicone Oil at Different Heating Rates

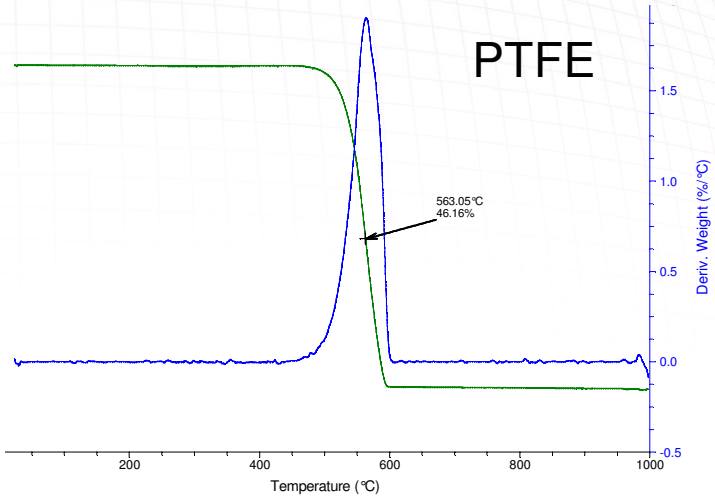
Chromatogram Plots



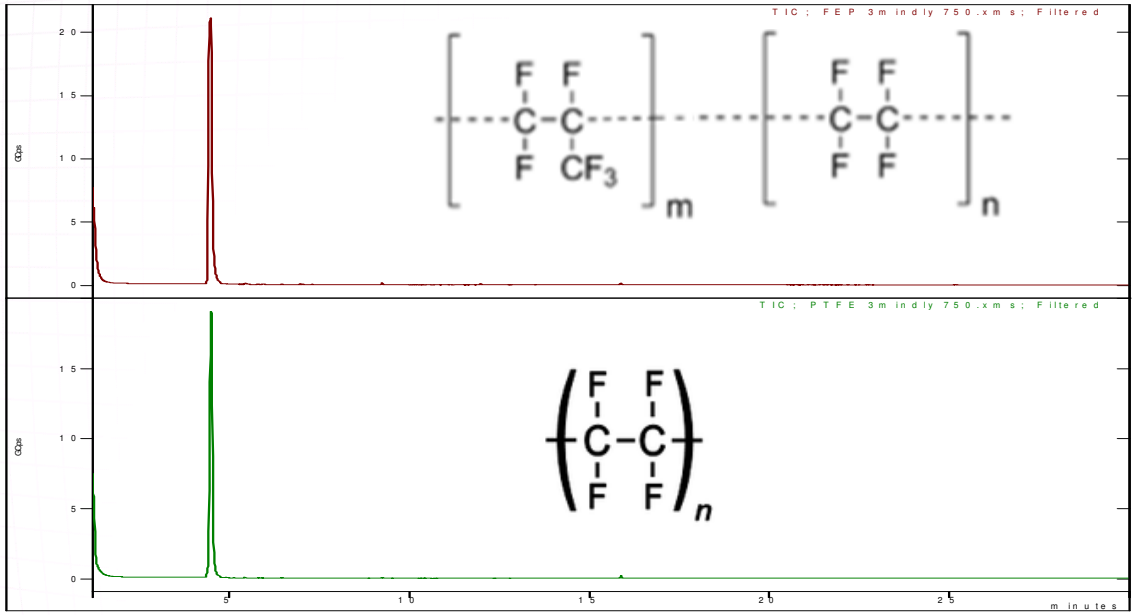
Pyrolysis provides insight into the TGA data. Pyrolysis indicates that silicone oil stays intact and simply boils off at heating rates of 30°C/min. The oil starts to display substantial fragmentation at 120°C

FEP Vs. PTFE Teflon

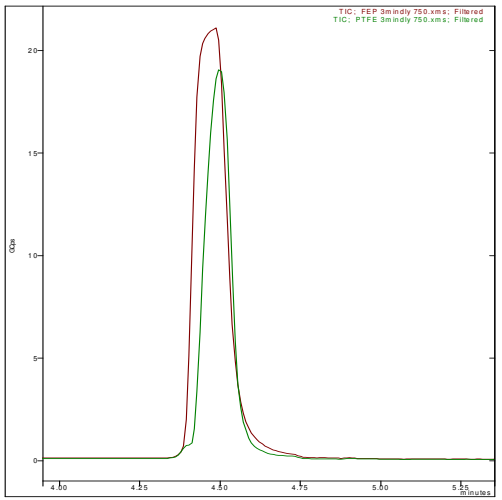




Chromatogram Plots

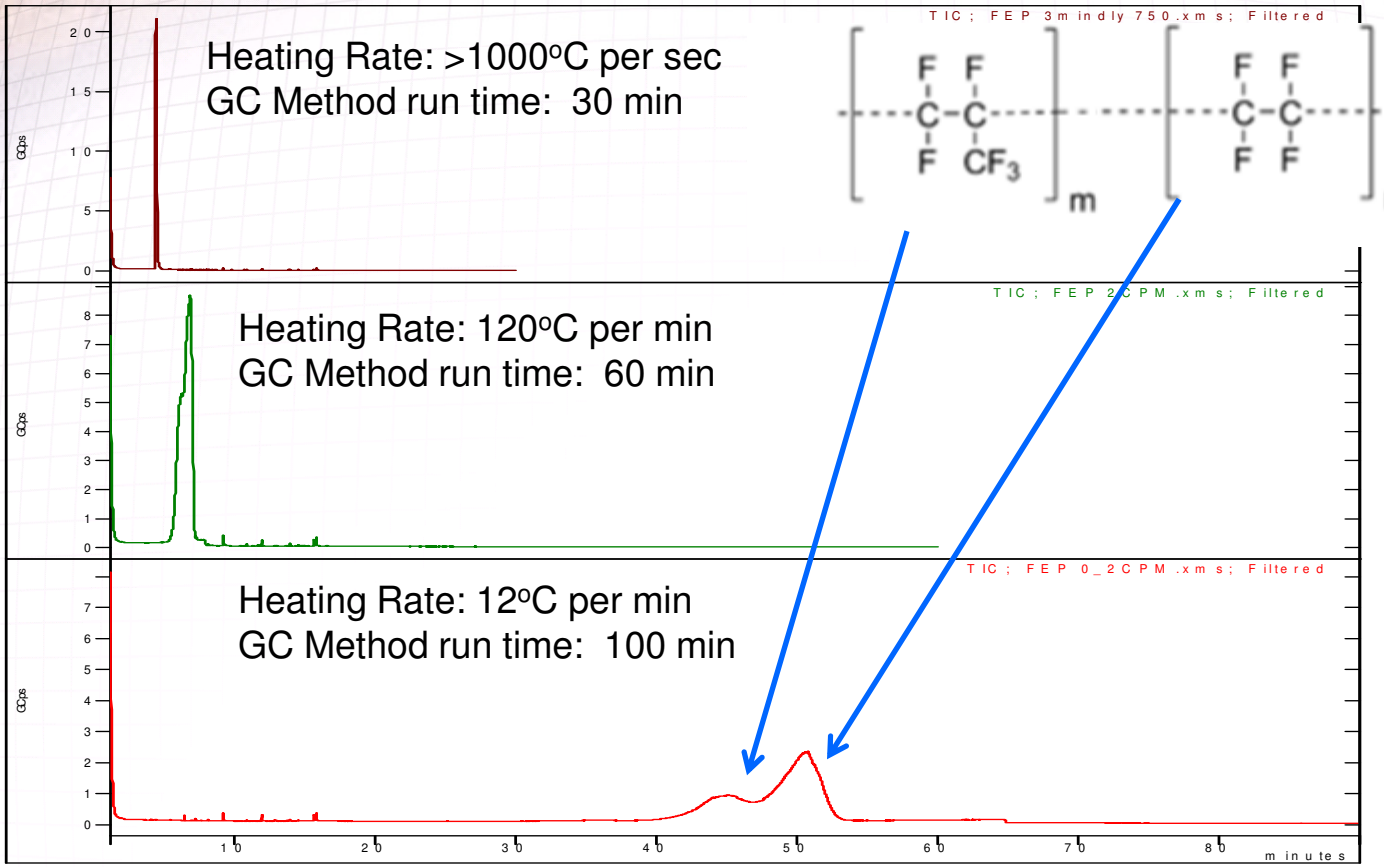


Overlaid Chromatogram Plots



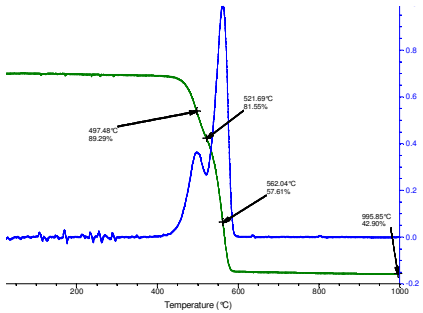
FEP Teflon Heated at Different Rates

Chromatogram Plots





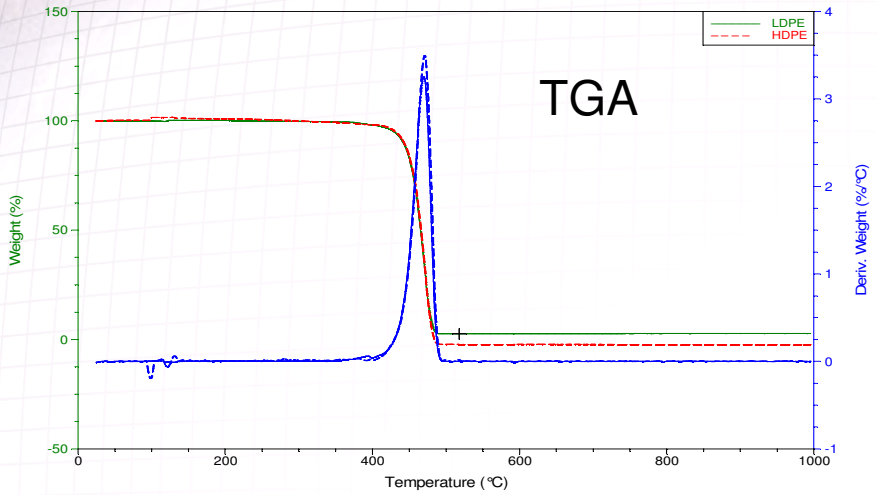
TGA

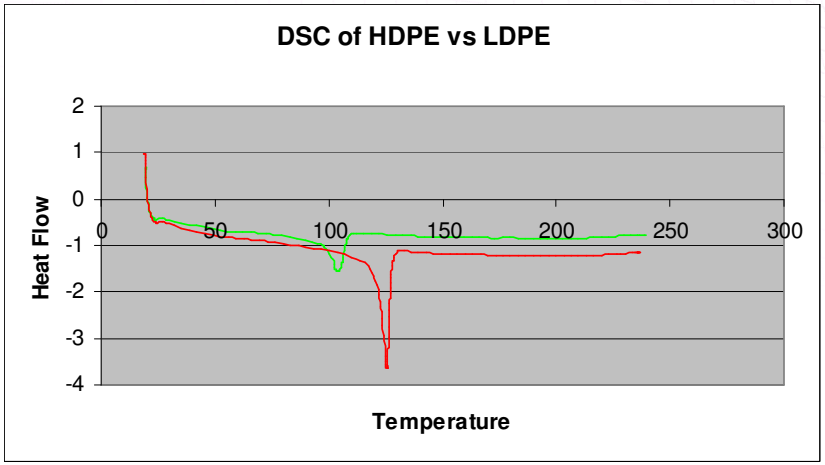


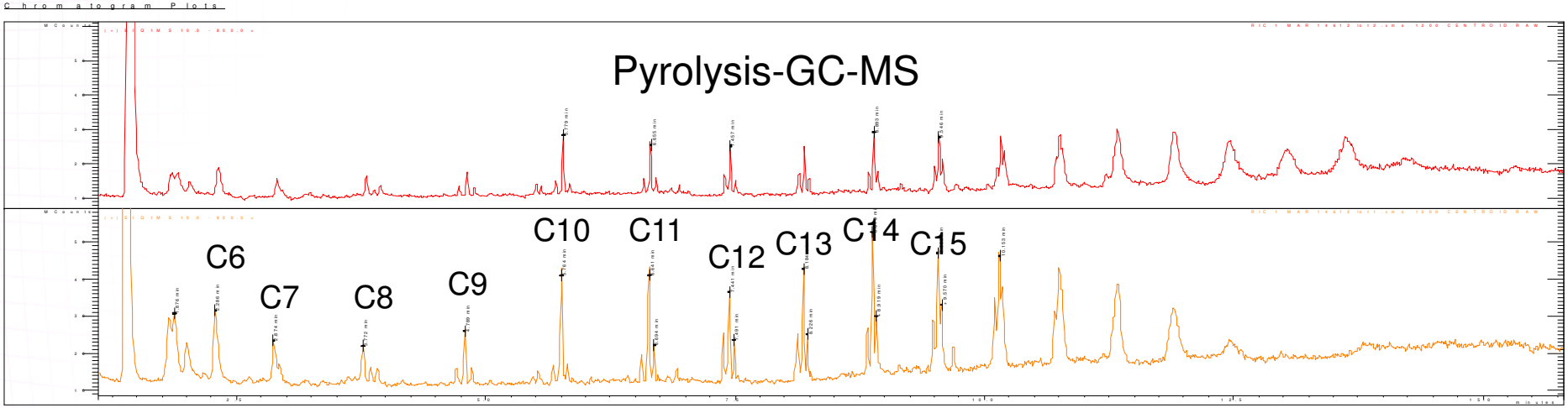
During pyrolysis, materials undergo thermal degradation via chemical pathways dictated by the thermal stability of the components. When pyrolysis is slowed to simulate TGA conditions, a thermal response pattern similar to what was observed with TGA first derivative plot is observed.

Thermal Analysis of HDPE and LDPE

Polyethylene: $-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-$

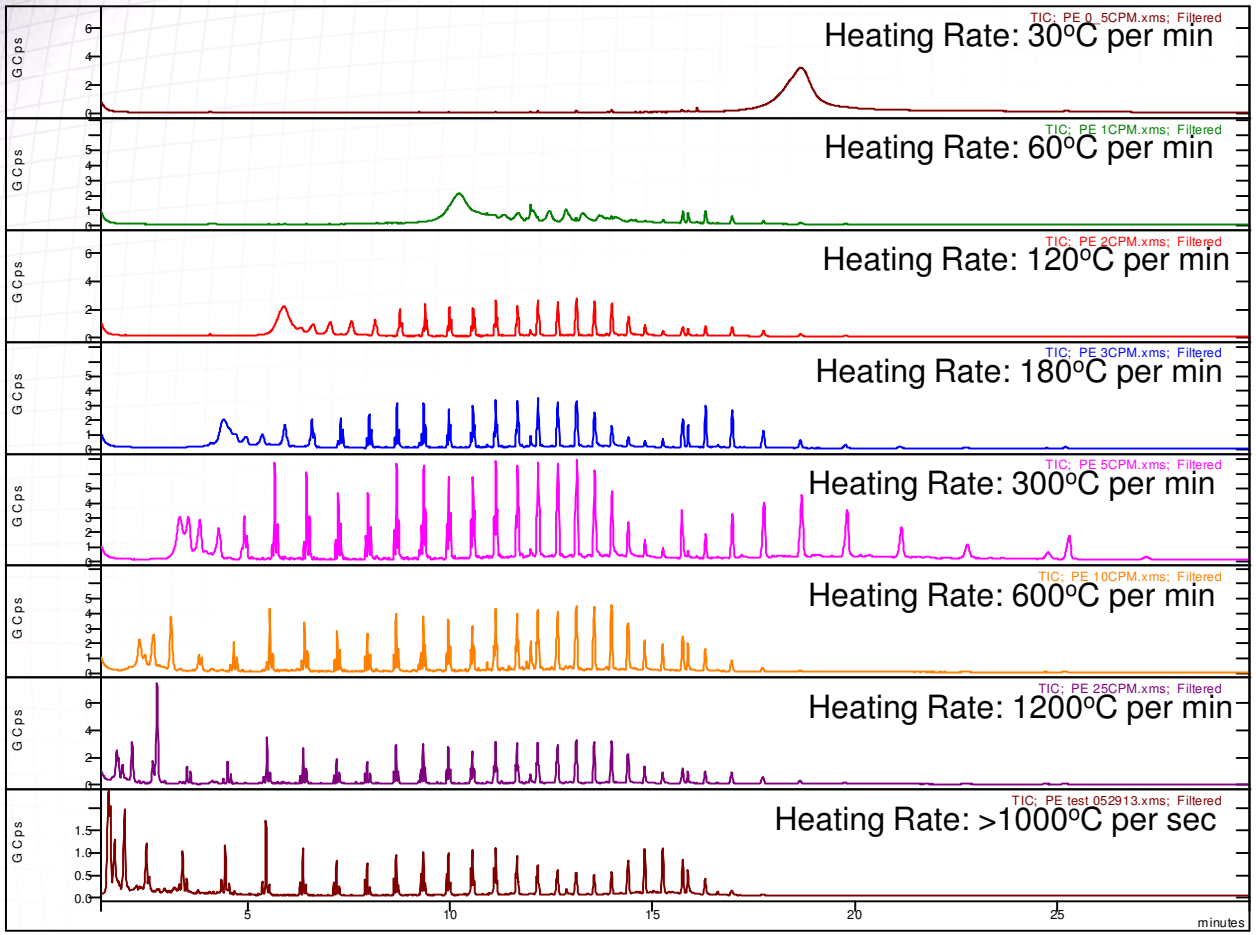






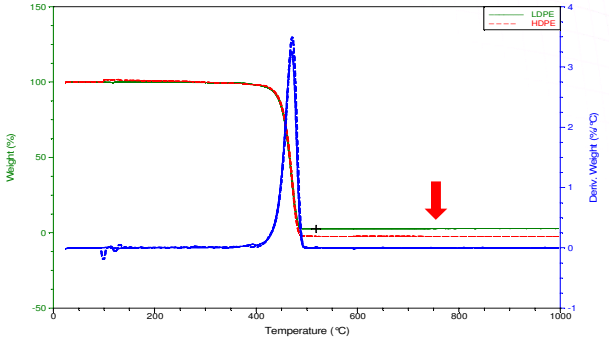
Temperature Ramp Pyrolysis

Heating PE in Pyrolysis chamber from 25°C to 750°C at different rates



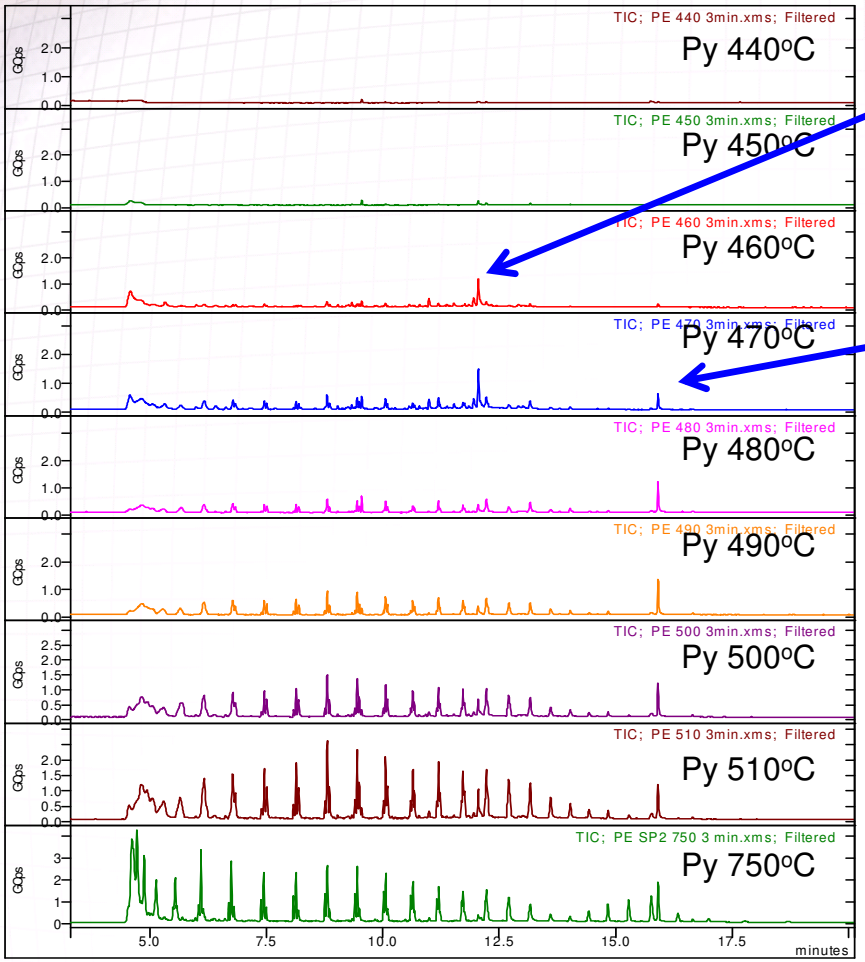
TGA

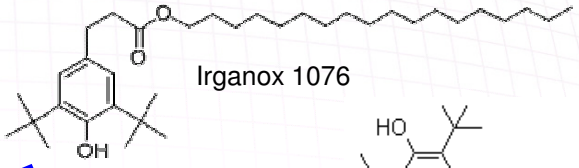
Heating Rate: 5°C per min



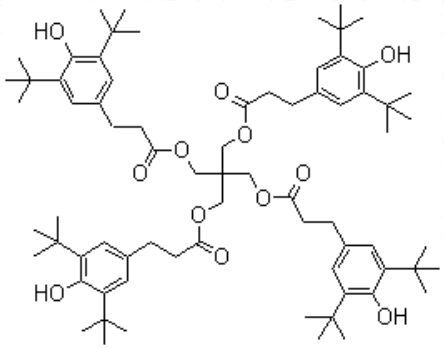
Correlating TGA and Pyrolysis Techniques

Chromatogram Plots

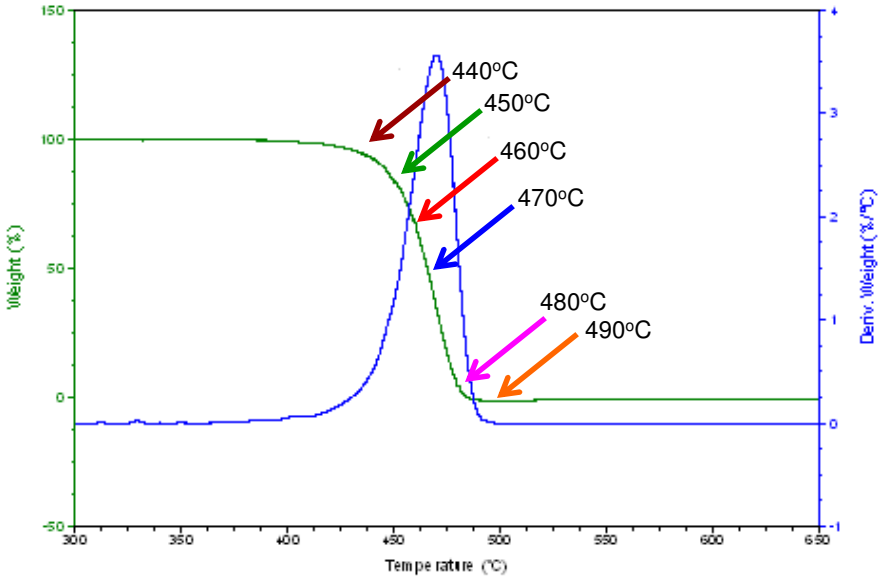




Irganox 1076



Irganox 1010

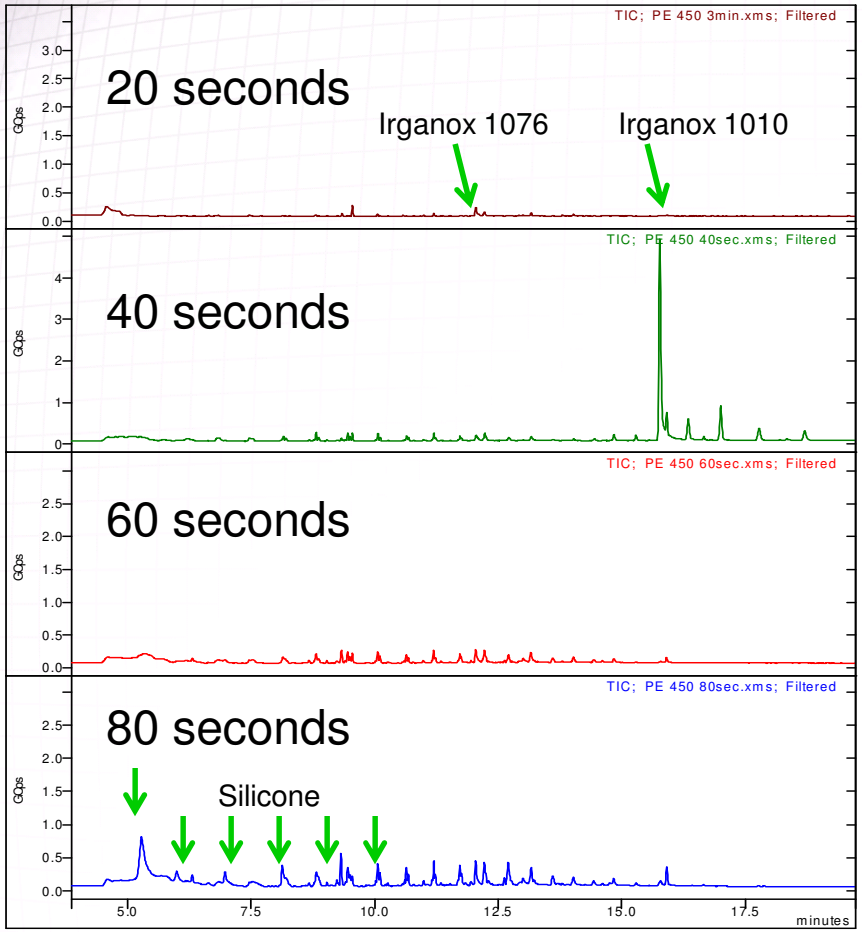


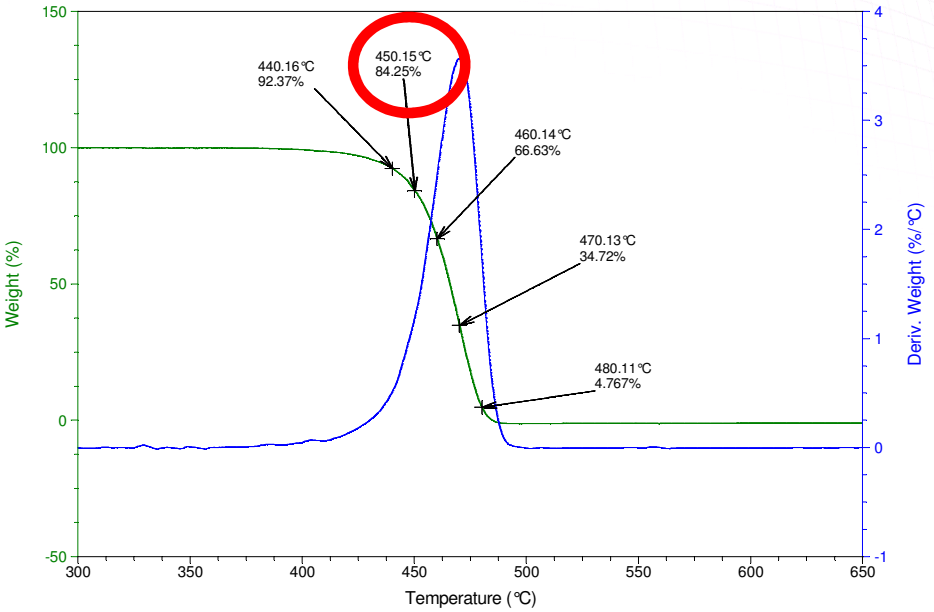
Pyrolysis at specified temperatures for 20 seconds

Thermal Analysis of PE

Pyrolysis at 450°C For Specified Duration

Chromatogram Plots

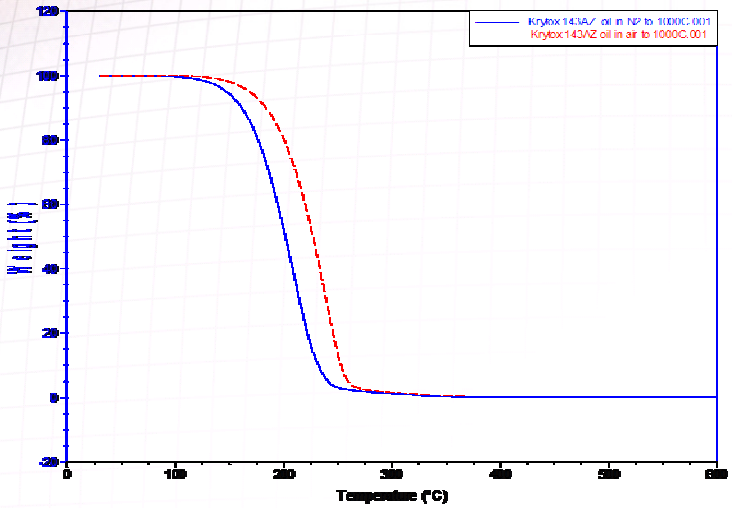




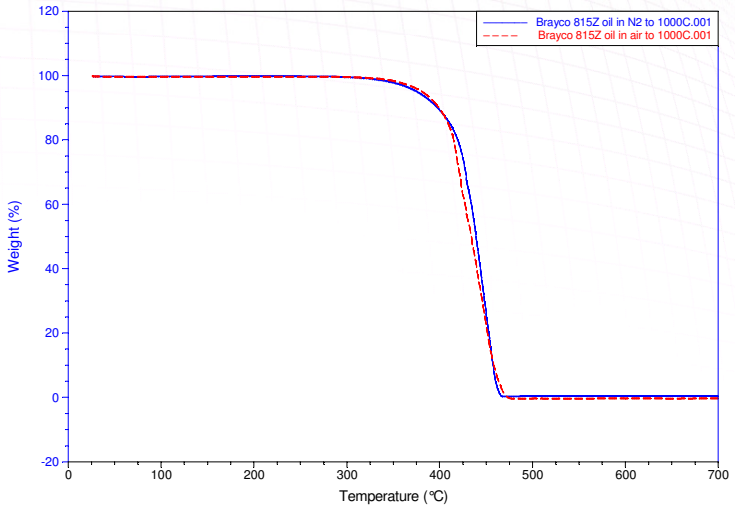
Modification of the thermal parameters at the onset of TGA degradation for PE can provide valuable information about the additives or contaminants.

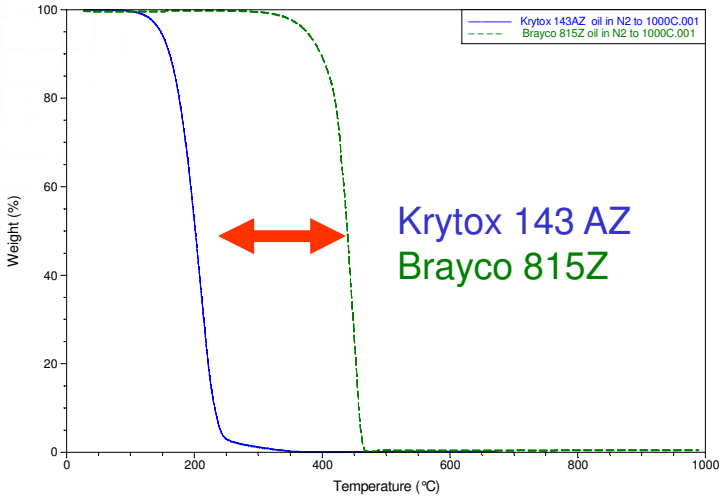
TGA Analysis of Fluorinated Materials

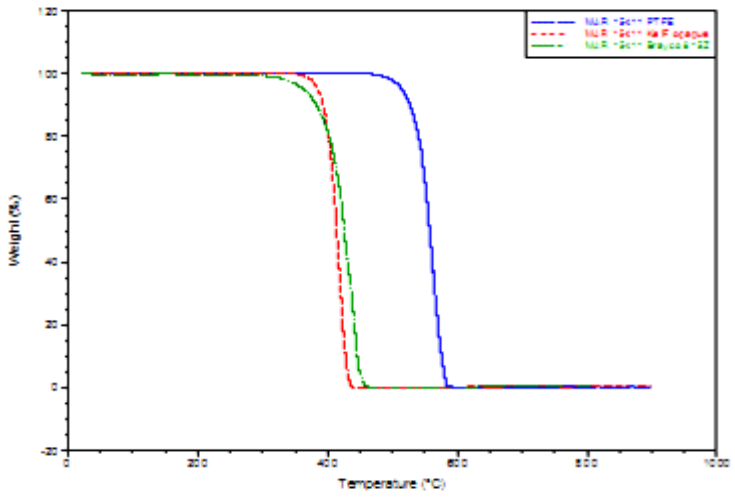
Krytox 143 AZ



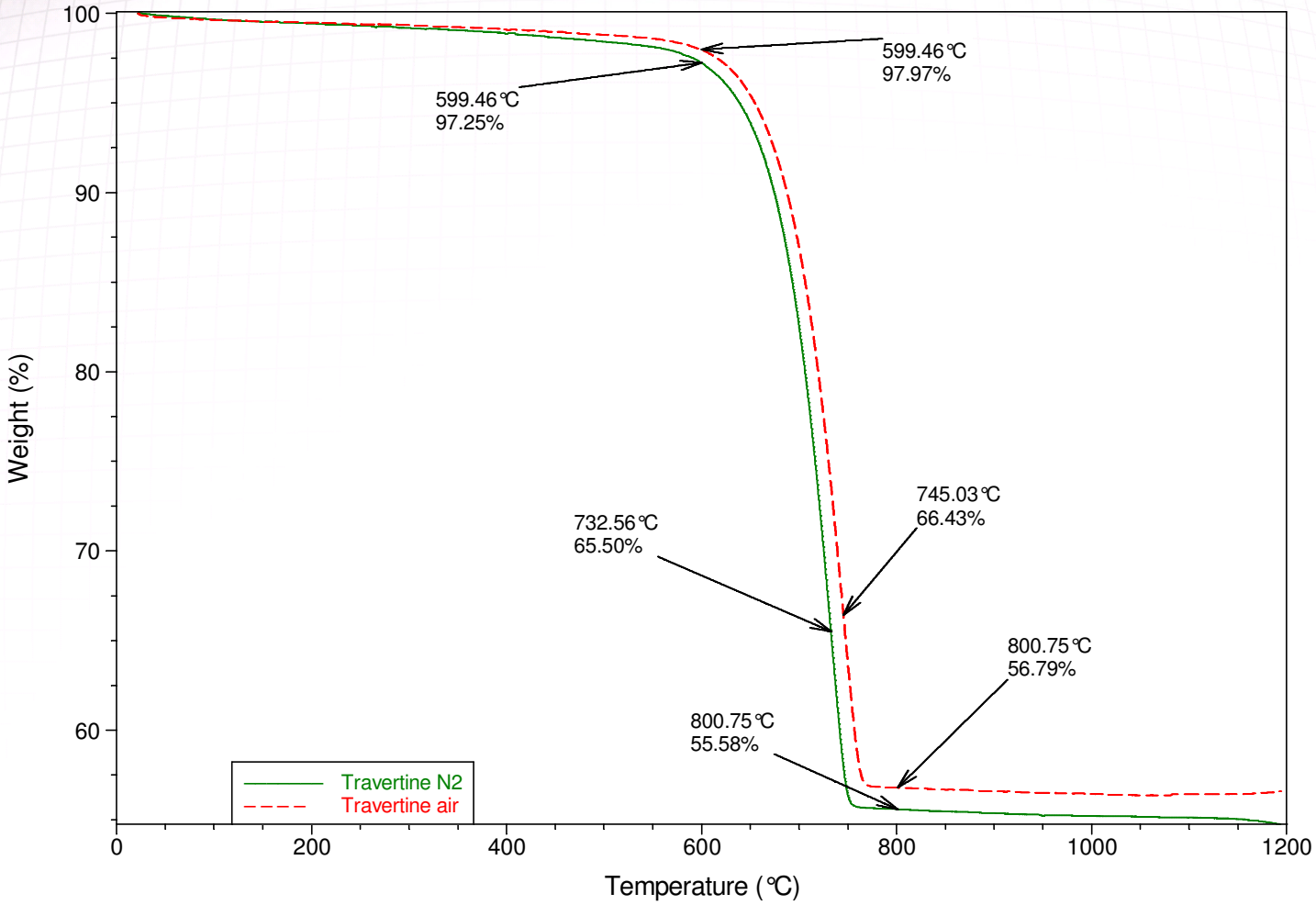
Brayco 815Z



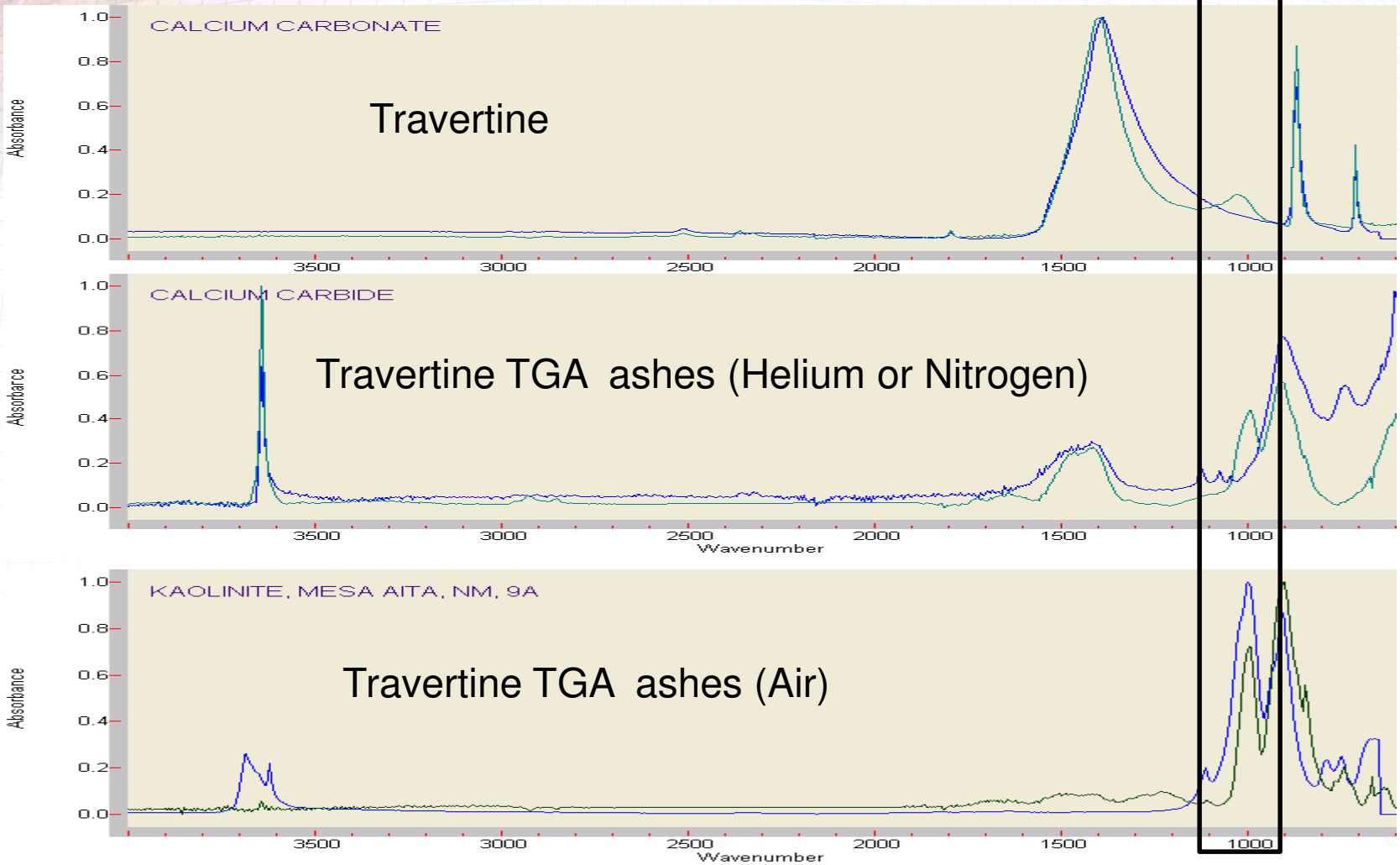




Thermal Response of Travertine in Different Atmospheres

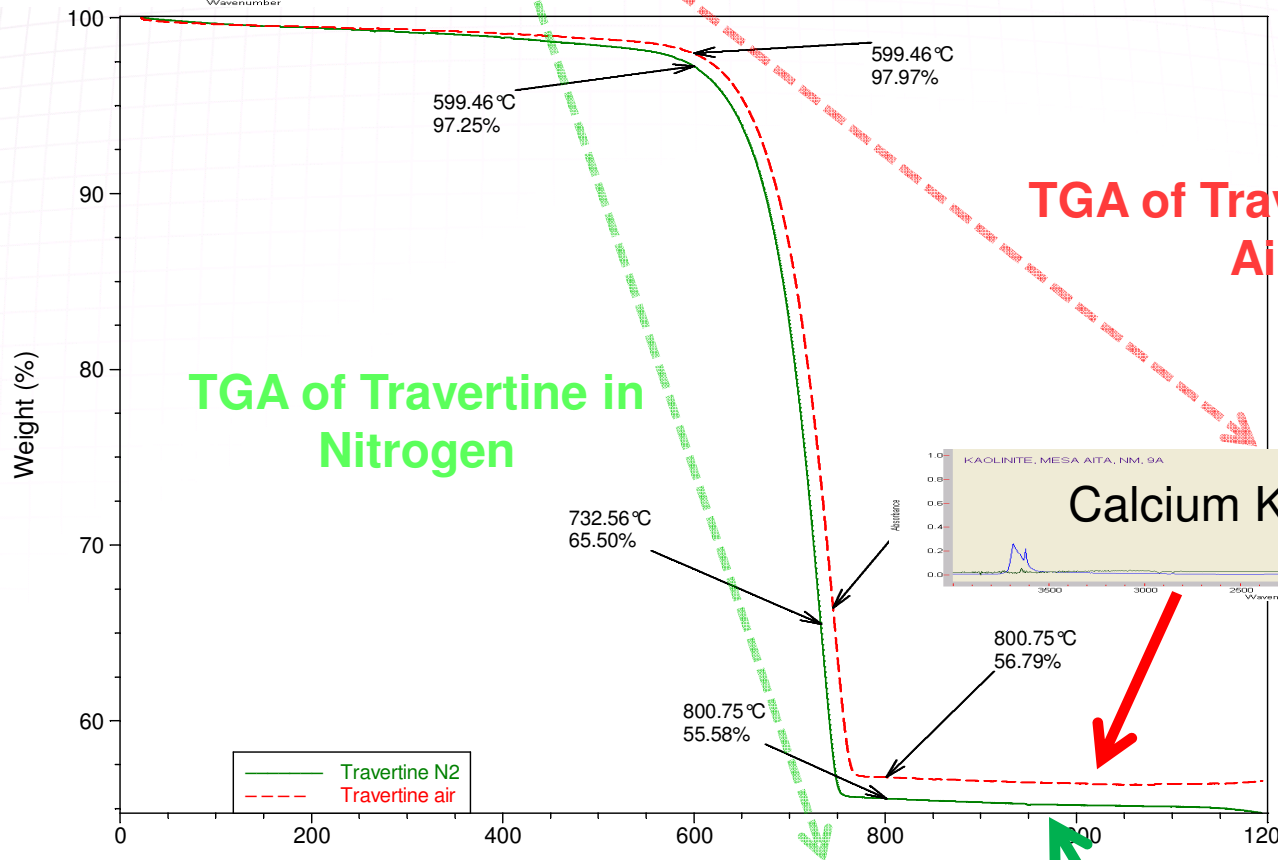


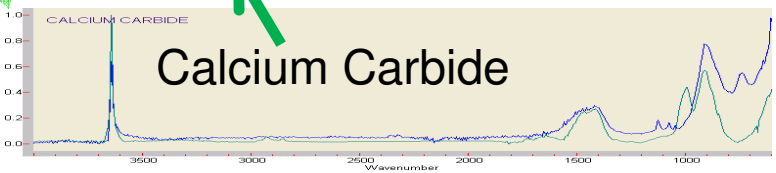
$CaCO_3 \longrightarrow CaC_2$ or Calcium Kaolinite



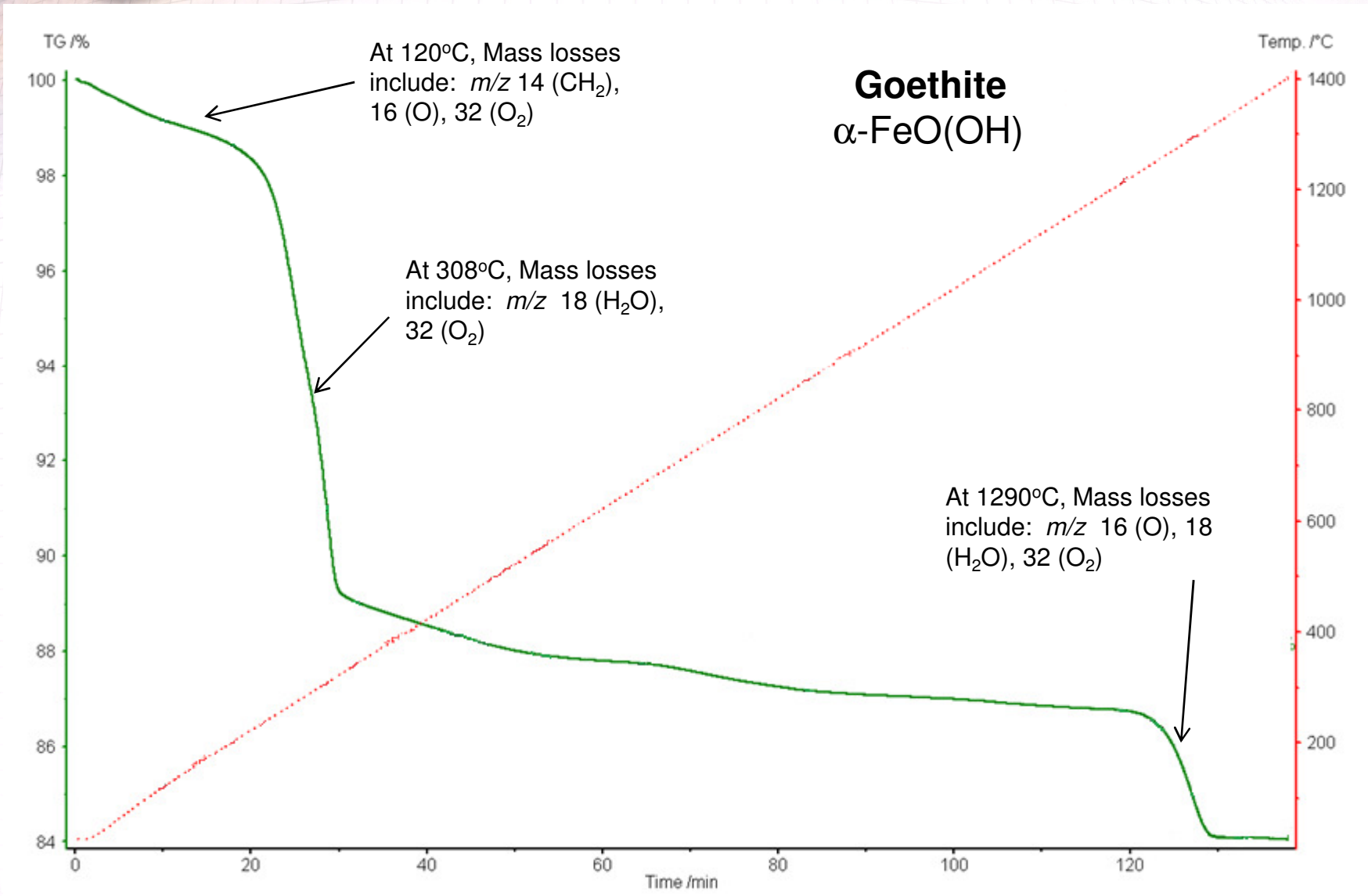
The Role of Gaseous Atmosphere During Thermal Decomposition of Travertine



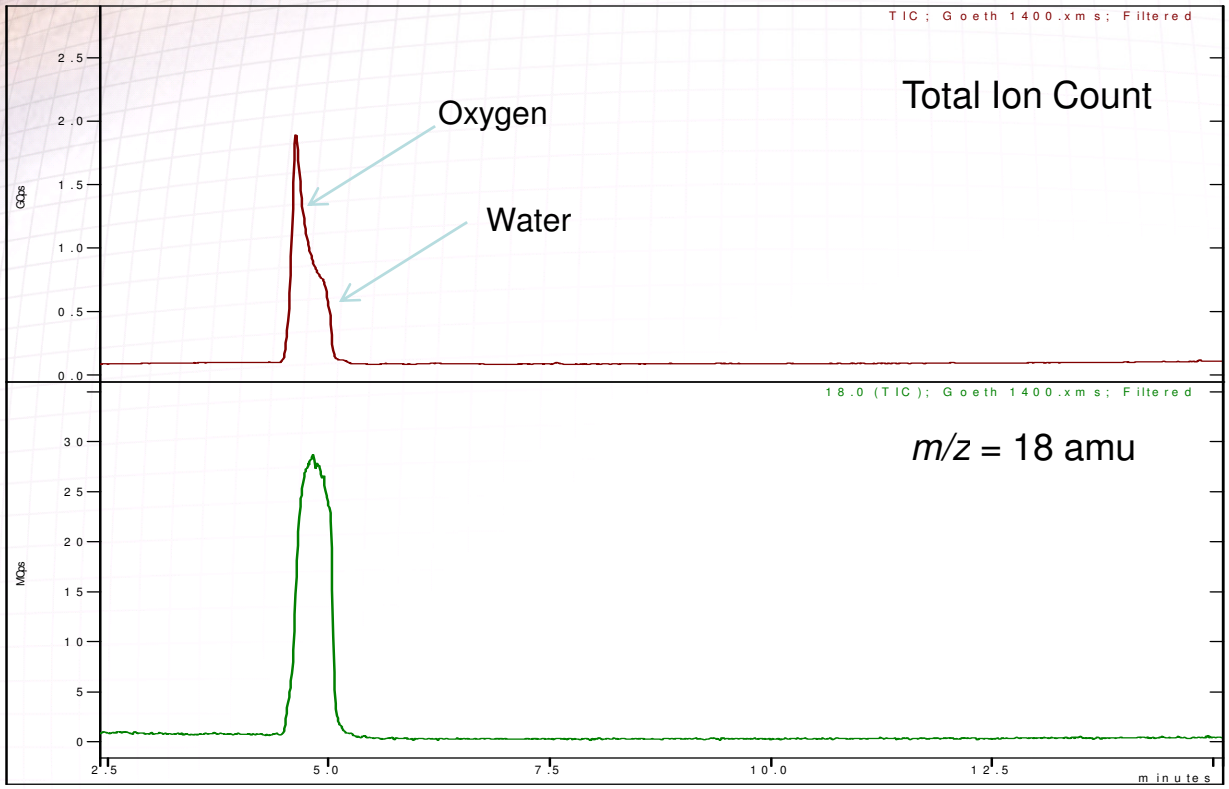




TGA Analysis of Geothite in Helium



Goethite Analysis by Py-GC-MS at 1400°C





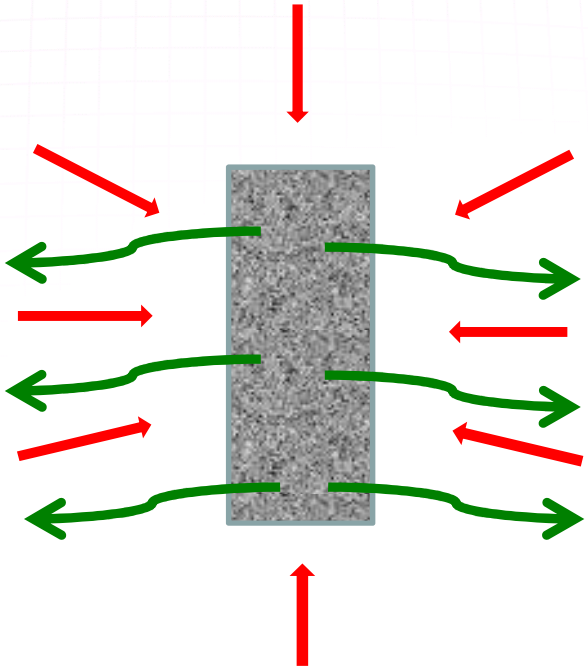


A sample of Goethite was first pyrolyzed at 750°C to remove all but the pertinent high temperature species. The same sample was then pyrolyzed at 1400°C to reveal two key molecules, Oxygen and Water. Since ample quantities of Goethite have been detected on the surface of Mars by Spirit, the NASA rover, we essentially have a source of water and oxygen waiting on Mars; we just have to heat the Goethite under the proper conditions to release these vital substances.

Applying Thermal Energy to Extract Chemical Information

Using Thermal Energy:

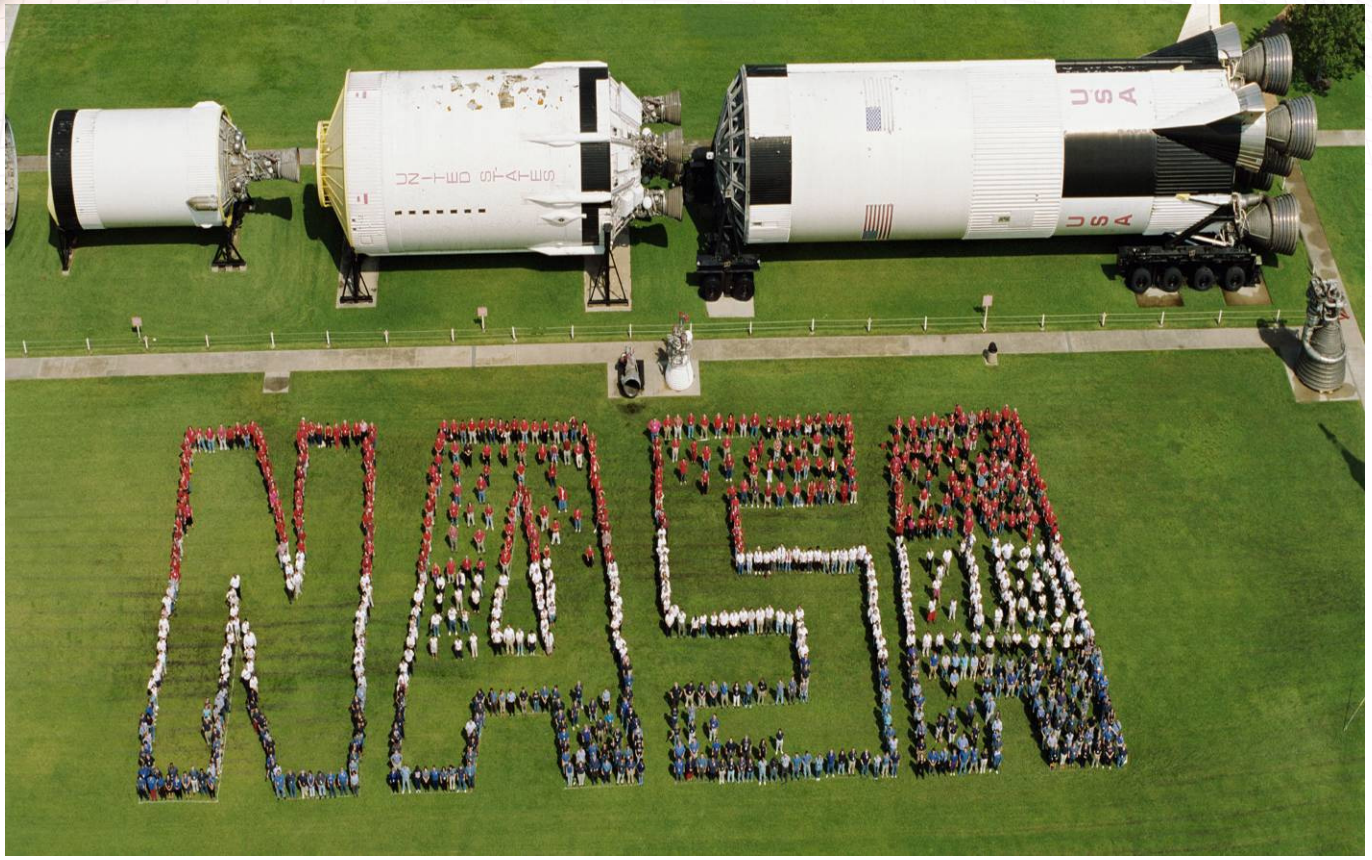
- How much Thermal Energy do we add
- How fast do we add the Thermal Energy
- How long do we maintain the Thermal Energy
- What atmosphere do we use
- How much sample do we use



Chemical Information

- Trapped solvent
- Organic additives
- Contaminants
- Labile Functional Groups
- Monomer identification
- Off-gassing information
- Inorganic additives

TGA DSC Pyrolysis-GC-MS TGA-MS-IR



Questions?

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