Pyrolysis Mass Spectrometry (PYMS)

Why should I use PYMS and why is it important?

- You have an unknown polymeric material and you need to definitively identify its composition in terms of starting monomers.
- You need to determine the ***potential additives*** in your polymer that comprise < 0.2% of the total.
- You may only have a very small sample of your polymeric material (~1 mg)

PYMS is one of the most versatile methods available today for unknown polymer identification. Jordi Labs has extensive experience in PYMS analysis and has developed proprietary libraries covering hundreds of known polymer systems and their additives.

How does PYMS work?

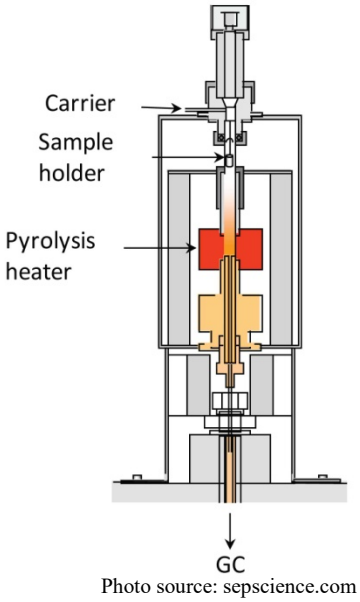

Photo source: sepscience.com

Sample Introduction and Pyrolysis: During PYMS, the sample (0.01 to 0.1 mg) is placed into a specialized metallic sample cup. This cup is then transferred into a pyrolysis chamber and subjected to ramped temperature heating. While the sample is heated, volatile and semi-volatile components are initially desorbed. This is followed by pyrolysis of less volatile materials. During pyrolysis, polymers "fragment" into monomeric and oligomeric components. The desorbed components are cryofocused on the instrument and then rapidly heated for transfer to a Gas Chromatogram (GC) column.

GC Separation and Detection: After cryofocusing, all of the sample components produced from the heating and pyrolysis steps are rapidly introduced to a capillary GC column. While traveling through the GC column, components are separated from each other mainly based on their boiling points. A typical GC column is 30 m long, and as each component elutes at the end of the column it is identified using an MS detector. A spectrum of each unknown component is produced and it is compared to libraries of compounds with known spectra (e.g., the EPA/NIH/NIST library of ~800,000 compounds). A "best match" is then determined for the unknown component.

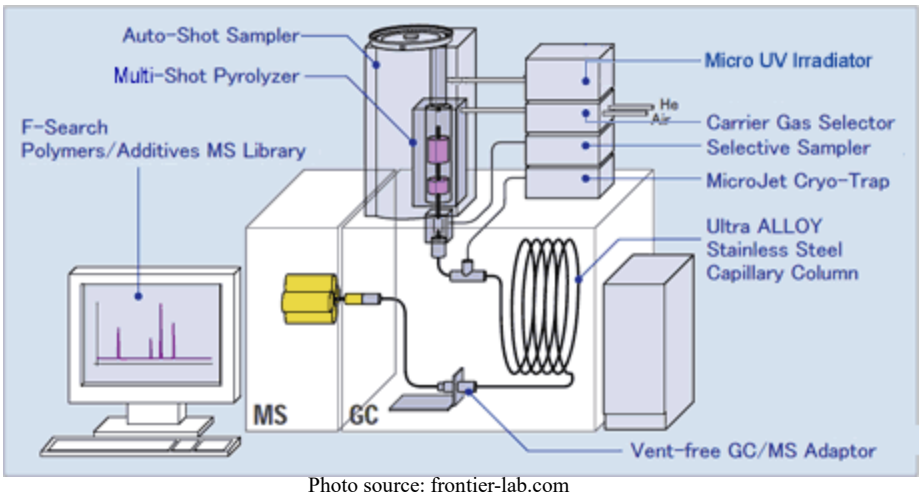

Photo source: frontier-lab.com

PY1 and PY2 (Double Shot Technique): Following the completion of the 1st pass analysis (PY1), the remaining portion of the sample in the cup is again heated (PY2) above its

decomposition temperature to analyze fragments that generate at much higher temperatures (up to 800 °C). The fragments (components) are again separated on a GC column and identified in the MS detector to determine their "best match".

Example Application: Analysis of a Nylon-6/Polyethylene Thin Film

The PYMS 1st pass chromatogram is characterized by a relatively intense peak identified as caprolactam (Nylon 6 monomer). The caprolactam dimer is also observed. In addition, the common polymer additive, Tinuvin 328, is detected. TINUVIN 328 is a UV absorber of thehydroxyphenylbenzotriazole class designed for coatings. Because of its extended UV absorption, TINUVIN 328 provides efficient protection to coatings and light sensitive substrates. The other peaks in the chromatogram are linear alkanes, consistent with pyrolysis fragments produced from polyethylene.

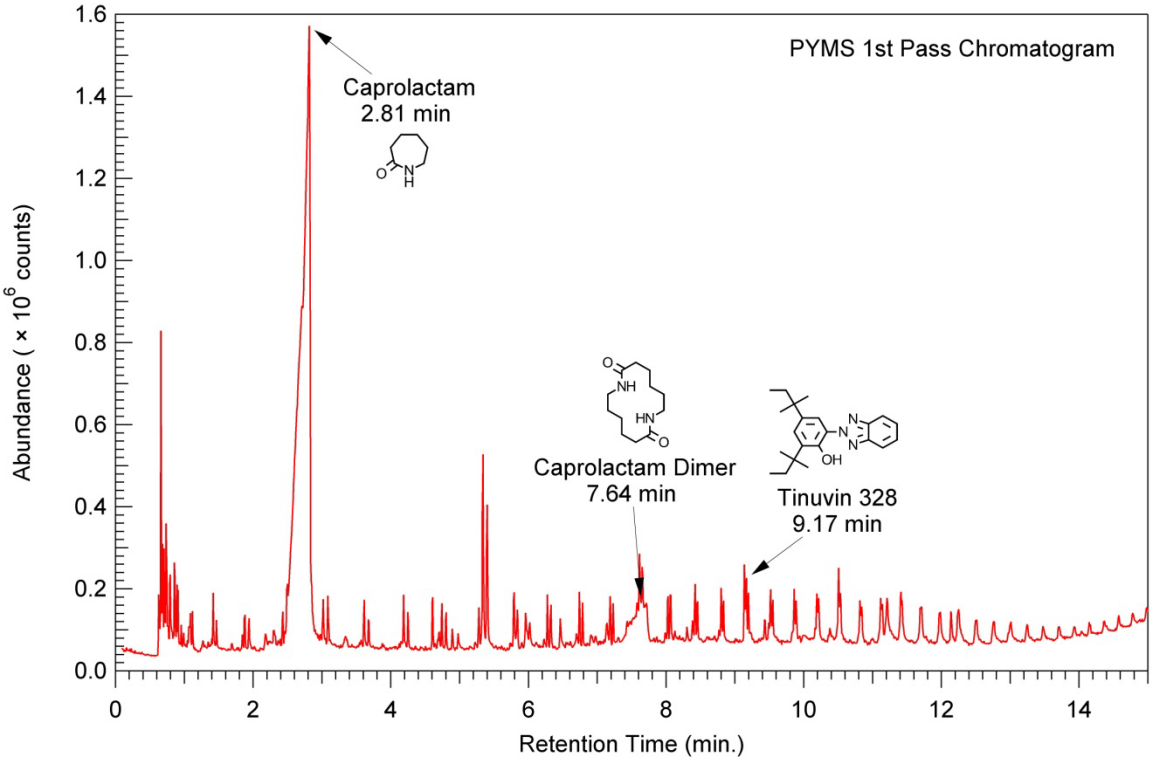

Figure 1: PYMS 1st pass Total Ion Chromatogram showing monomer Caprolactam, Caprolactam Dimer, and Tinuvin 328

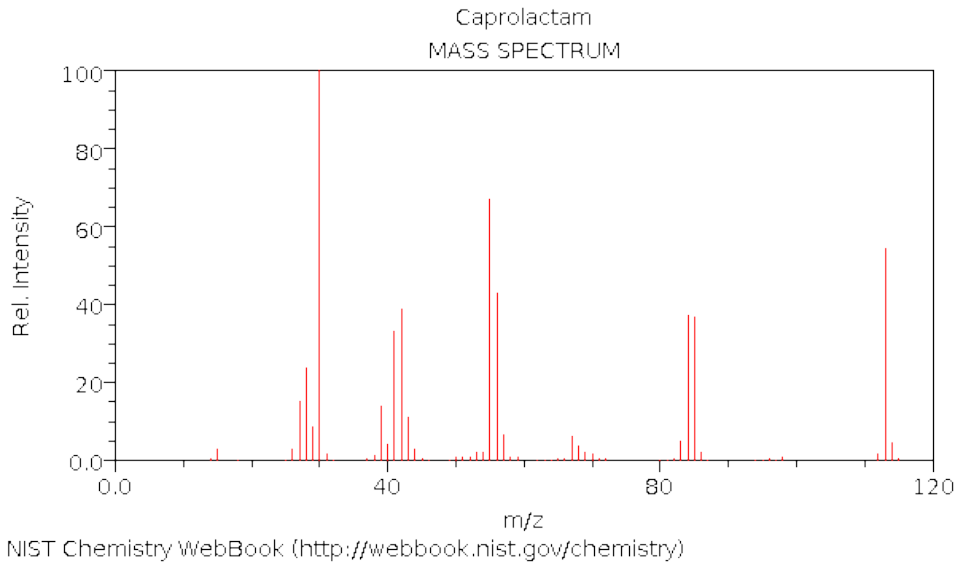

Figure 2: Example Mass Spectrum for Caprolactam

The PYMS second pass chromatogram shows a large number of peaks with the pattern characteristic of a series of linear alkanes. The mass spectra observed are all consistent with various linear alkane fragments. This is the typical behavior of polyethylene. The polyethylene fragments dominated the chromatogram of both the 1st and 2nd heating passes. This suggests that the material is a polymer blend comprised of polyethylene as the major component with a smaller amount of Nylon-6. The material is stabilized with Tinuvin 328.

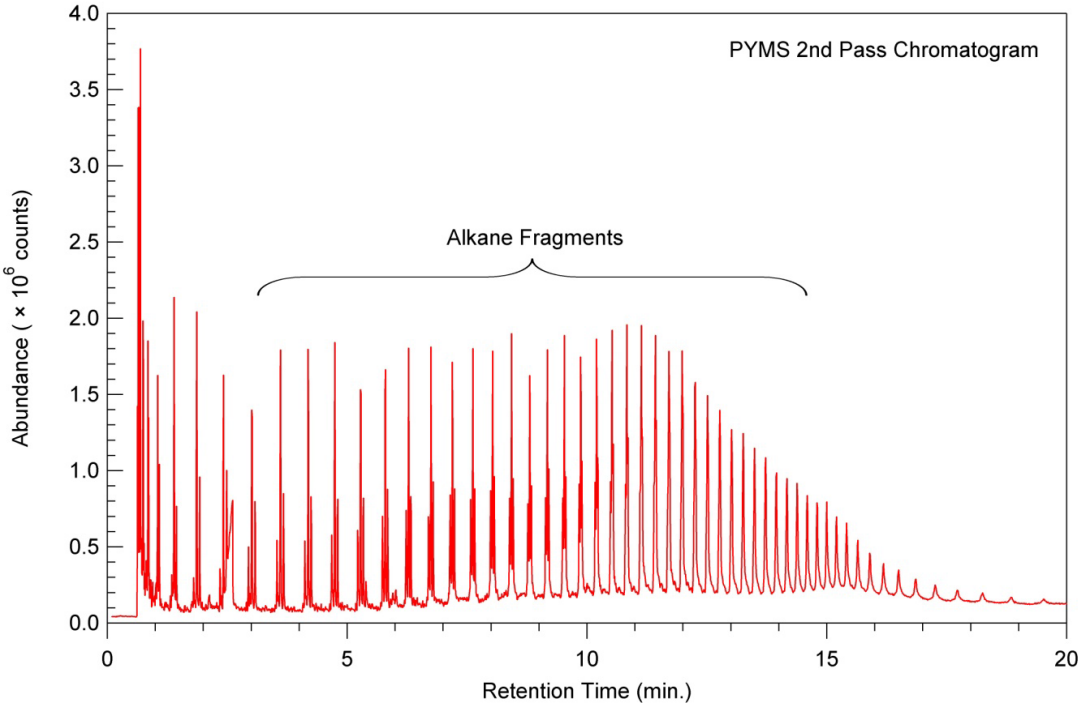

Figure 3: PYMS 2nd pass Total Ion Chromatogram showing Alkane Fragments