

**Application Note****Fibers****Author:**

T. Wampler

Analytical pyrolysis is the process of using heat energy to break the molecular bonds in large molecules, creating smaller, volatile compounds which can then be analyzed by common techniques such as gas chromatography, infrared spectrometry, and direct mass spectrometry. Since the bonds in each molecule break in a predictable way, depending on their energy, a pattern of small molecules results which can be used to identify the original material. Polymers are particularly suitable for analysis by pyrolysis because their large size makes them difficult to analyze. Pyrolysis-GC has been used for decades in the analysis of synthetic polymers such as polyolefins, PVC, synthetic rubbers, etc., including the study of materials used to make textile fibers (polyester, acrylics, and Nylon, to name a few).

The same methods may be used to investigate the properties and chemical nature of natural polymers and the fibers made from them. Plant fibers such as cotton, hemp, and flax are polymers of glucose in the form of cellulose. Animal fibers such as wool, angora, and silk are proteins. Upon pyrolysis, the particular sugars or amino acids that compose the polymer will break apart in predictable ways, and the original polymer can be easily identified from the breakdown products.

When heated to 700°C, cellulose undergoes a variety of degradation mechanisms, chiefly eliminating water and carbon dioxide, but producing a wide range of organic molecules as well. Figure 1 shows the chromatogram that results from the pyrolysis of cotton thread at 700°C for ten seconds.

The proteins found in wool and silk produce completely different pyrolysates when heated. Examples of this are shown in Figures 2 and 3. Here, the major fragments are aromatics and phenols generated from the pyrolysis of the amino acids tyrosine and phenylalanine, as well as some smaller fragments.

Because each biopolymer pyrolyzes to produce its own characteristic fingerprint of pyrolysis volatiles, these fibers are easy to distinguish from each other. The content of fiber blends may also be determined using this technique. When a textile fiber is comprised of more than one type of polymer, each of the polymers still pyrolyze to produce its characteristic chromatographic peaks so that the nature of the blended material may be elucidated.

**Instrument Conditions**  
**Pyroprobe**

**Pyrolysis:**

Initial: 700°C 10 seconds

Ramp: 20°C/ mSec

**GC-FID**

Column: 50 x 0.25mm SE-54

Carrier: Helium, 60:1 split

Injector: 300°C

Oven: 50°C for 3 minutes  
then 8°C/min to 300°C

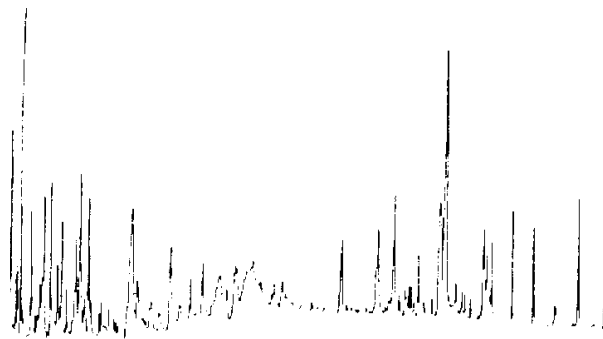


Figure 1. Pyrolysis of Cotton Thread

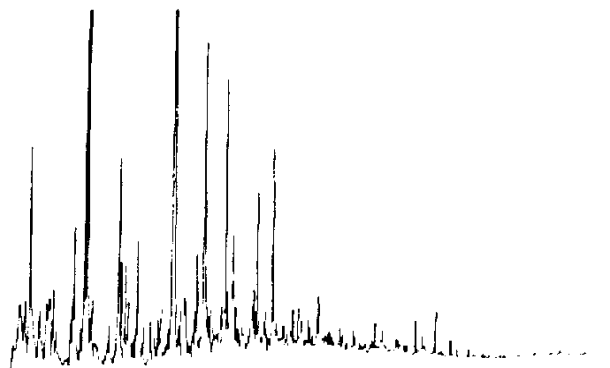


Figure 2. Pyrolysis of Raw Wool

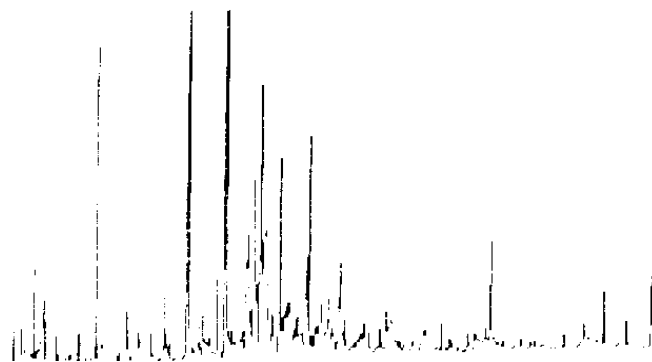


Figure 3. Pyrolysis of Silk

FOR MORE INFORMATION  
CONCERNING THIS APPLICATION, WE RECOMMEND THE  
FOLLOWING READING:

Kawaoka, K. "Characteristics of Polymers by Pyrolysis-Gas Chromatography-Mass Spectrometry", Proceeding of the International Symposium on the Analysis and Identifications of Polymers, FBI Academy, Quantico, VA, pp. 39-40.

Levy, E.J. and T.P. Wampler. "Optimized Biopolymer Pyrolysis GC Characterization", American Biotechnology Lab., Vol. 5, #1, pp. 56-60.