

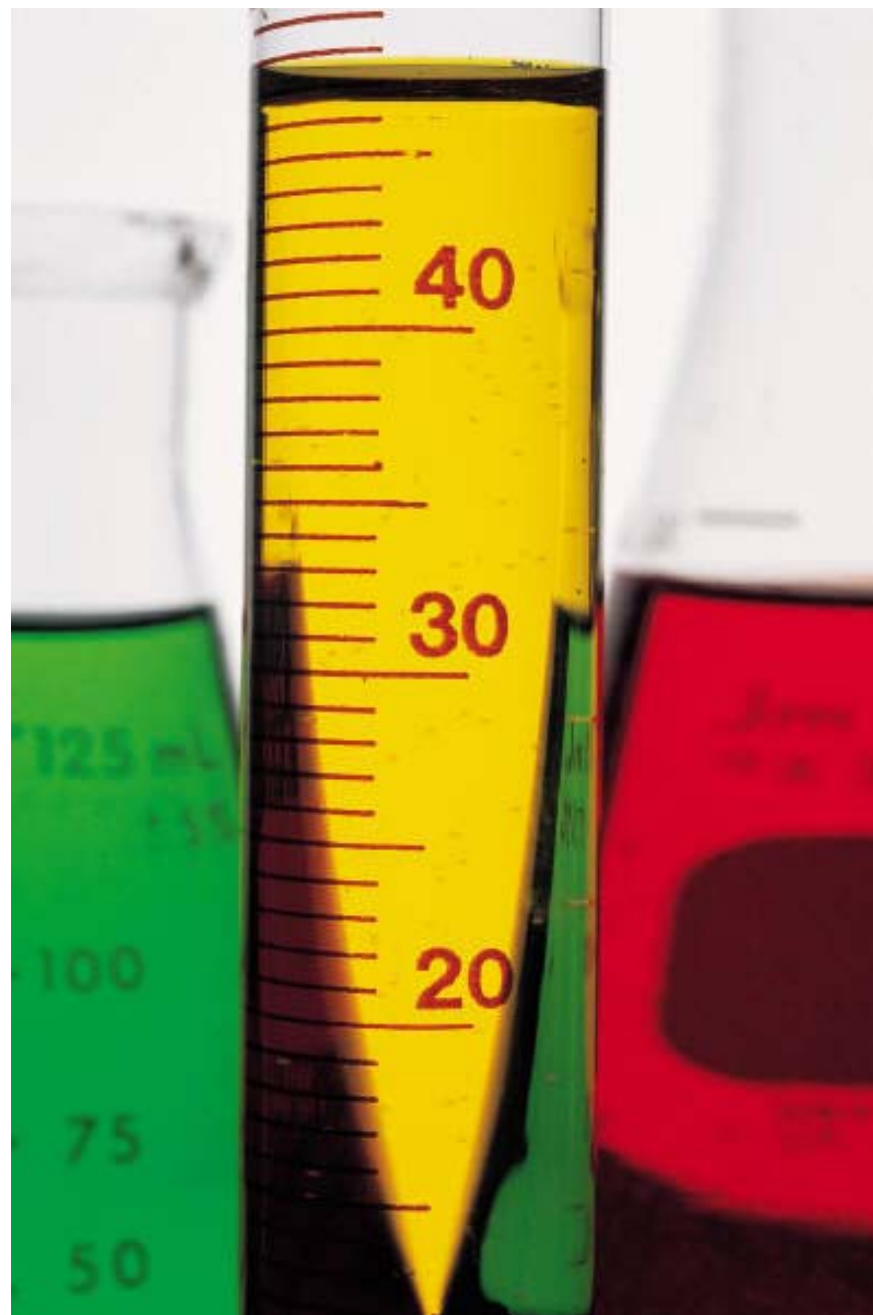
Real World Challenges in Plastic Analysis

Presented By:
Dr. Jack Hubball



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Why Is There a Need for a Industrial Additive Standards?

Good Science

- Analytical work relies on a comparison that is made with well-characterized standards
- Whether a laboratory prepares their standards in-house, or buys them from an outside source, there is always the need to know that they are prepared and identified correctly



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Why Is There a Need for a Industrial Additive Standards? (cont.)

- **Regulatory requirements**

- **ASTM – Method D6042-96**

Determination of Phenolic Antioxidants & Erucamide Slip Additives in Polypropylene Homopolymer Formulations Using Liquid Chromatography

- **ASTM – Proposed Method D7065-06**

P-tert-Octylphenol, Nonylphenol Monoethoxylate & Nonophenol Diethoxylate and Tech Nonylphenol, Mono- and Di-ethoxylate

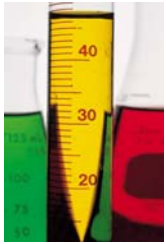
- **FDA**

- **EU Directives**



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Categories of Polymer Additives

- Antifoams
- Antioxidants
- Antistatic Agents
- Blowing Agents
- Coupling Agents
- Cross-Linking Agents
- Flame Retardants
- Lubricants and Related Additives
- Plasticizers
- Retarders
- UV stabilizers
- Colorants
- Release agents



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Chemical Additive Standards are different...

- Typically we think of a Chemical Reference Standard as a single pure analyte
- Typically a Reference Standard has a traceability link to a NIST SRM or other Primary Standard



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Chemical Additive Standards are different... (cont.)

- **Chemical Additives are often technical mixes of several different chemicals**
- **Purity may be less than 90%**
- **Characterization in a real world sample can be challenging**
- **Traceability can be dubious**



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Extraction Methods

- **Solvent Extraction**
 - Native material
 - Pulverized Polymer
- **Soxhlet Extraction**
 - Native material
 - Pulverized Polymer
- **Super Critical Fluid Extraction**
- **Microwave**
- **Thermal Desorption**



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Polymer Sample in GC Inlet

Thermal desorption of Polymer Additives



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Polymer Sample in GC Inlet with Aluminum Foil Plug



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Analysis

- **LC/MS**
 - Higher molecular weight capability
 - No thermal degradation
 - Poor resolution
- **GC/MS**
 - Better resolution
 - Possible thermal degradation
 - Lower molecular weight capability



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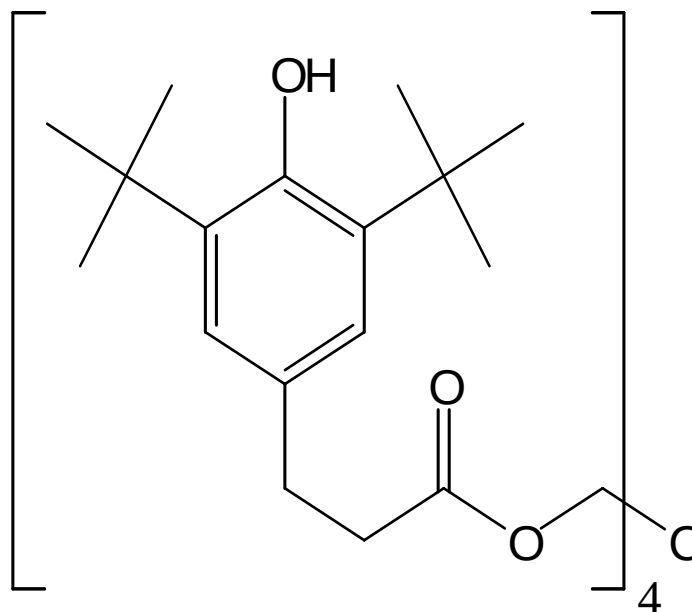
You can ID a compound by the Representative Fragments

Ethanox 310

- Penterythritol tetrakis (3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate)

MW = 1177.65

$C_{73}H_{108}O_{12}$

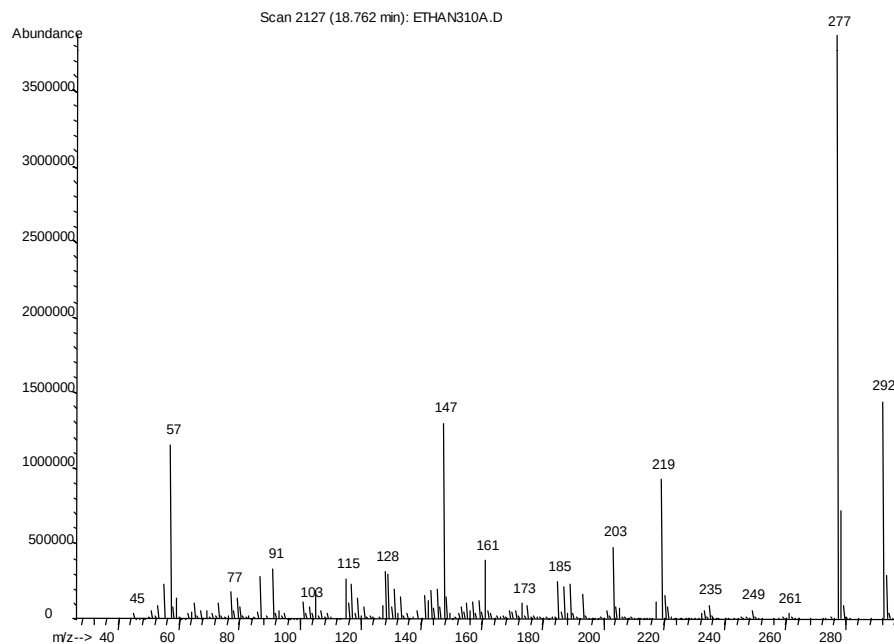
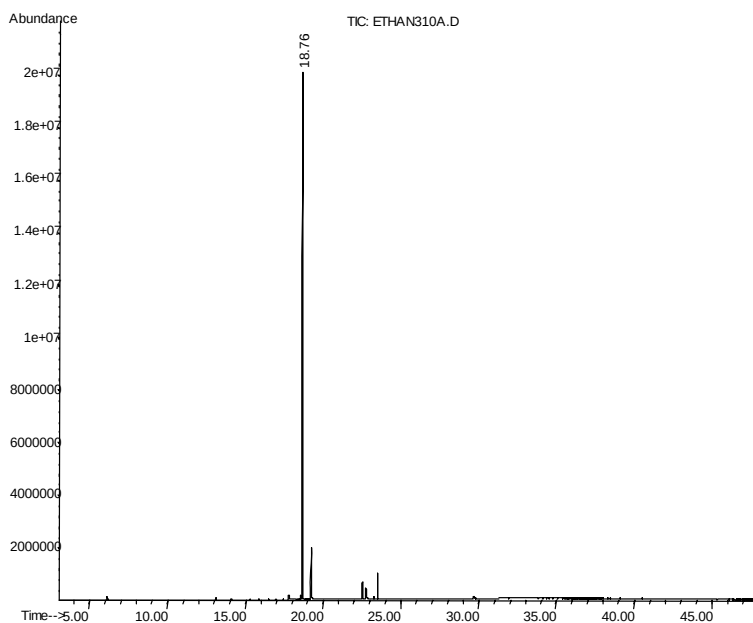


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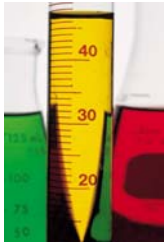
Ethanox 310 GC and Mass Spectrum of the Major Fragment



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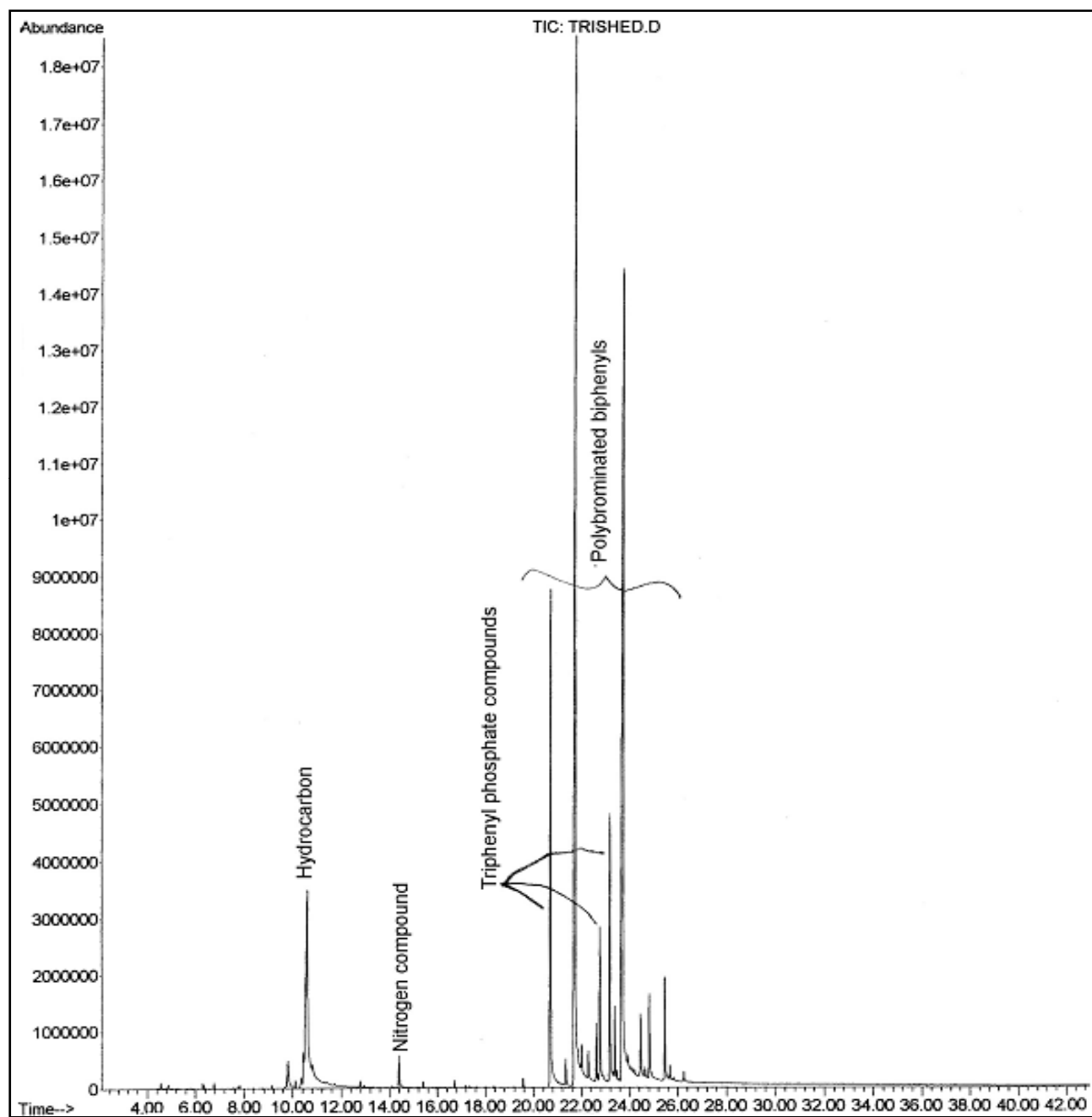


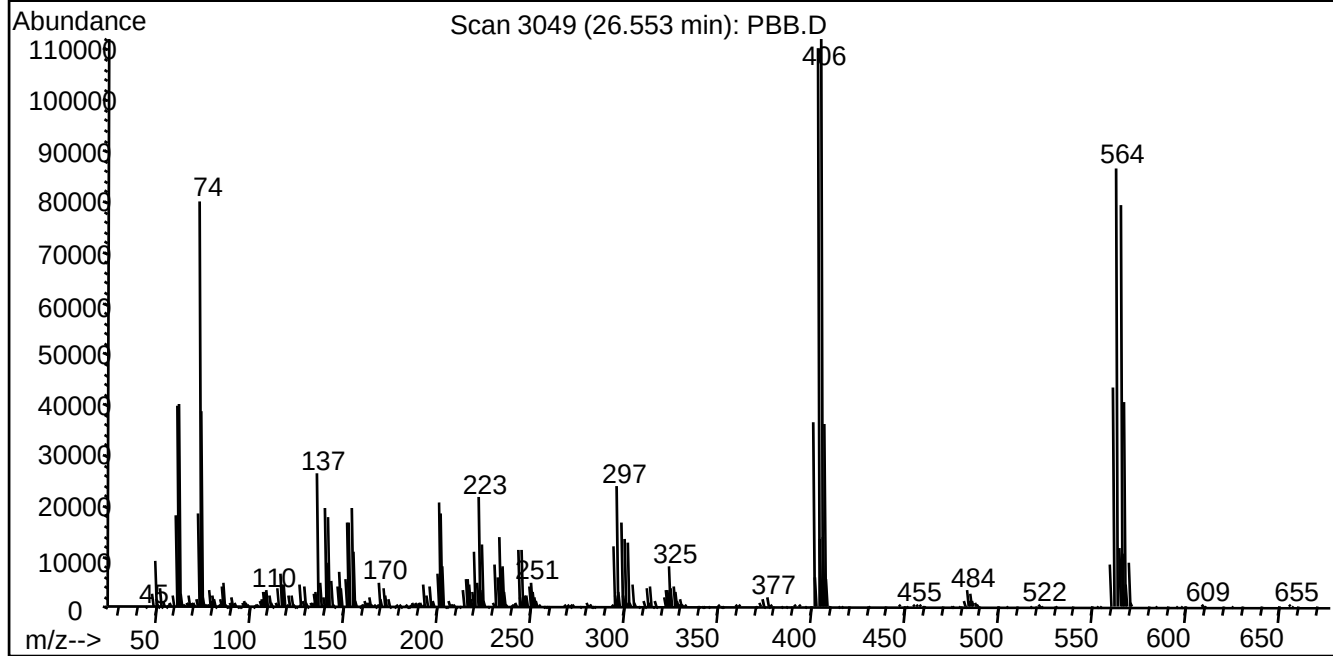
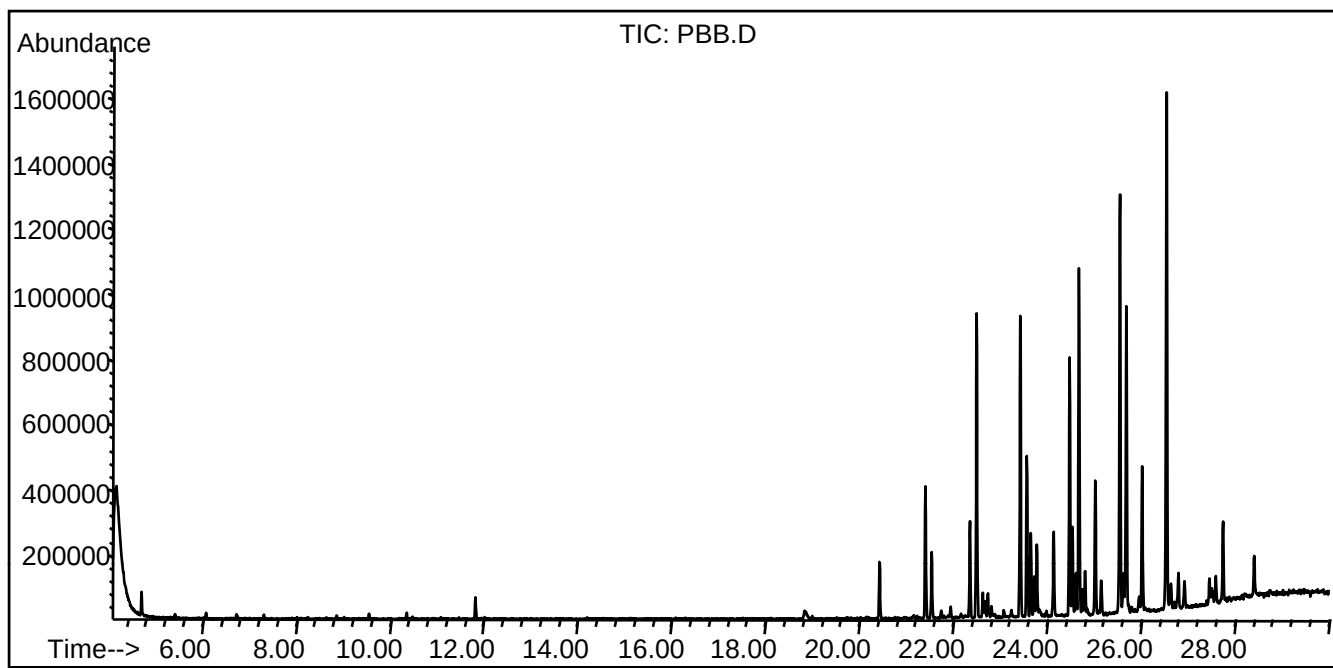
Insulators containing PBDEs



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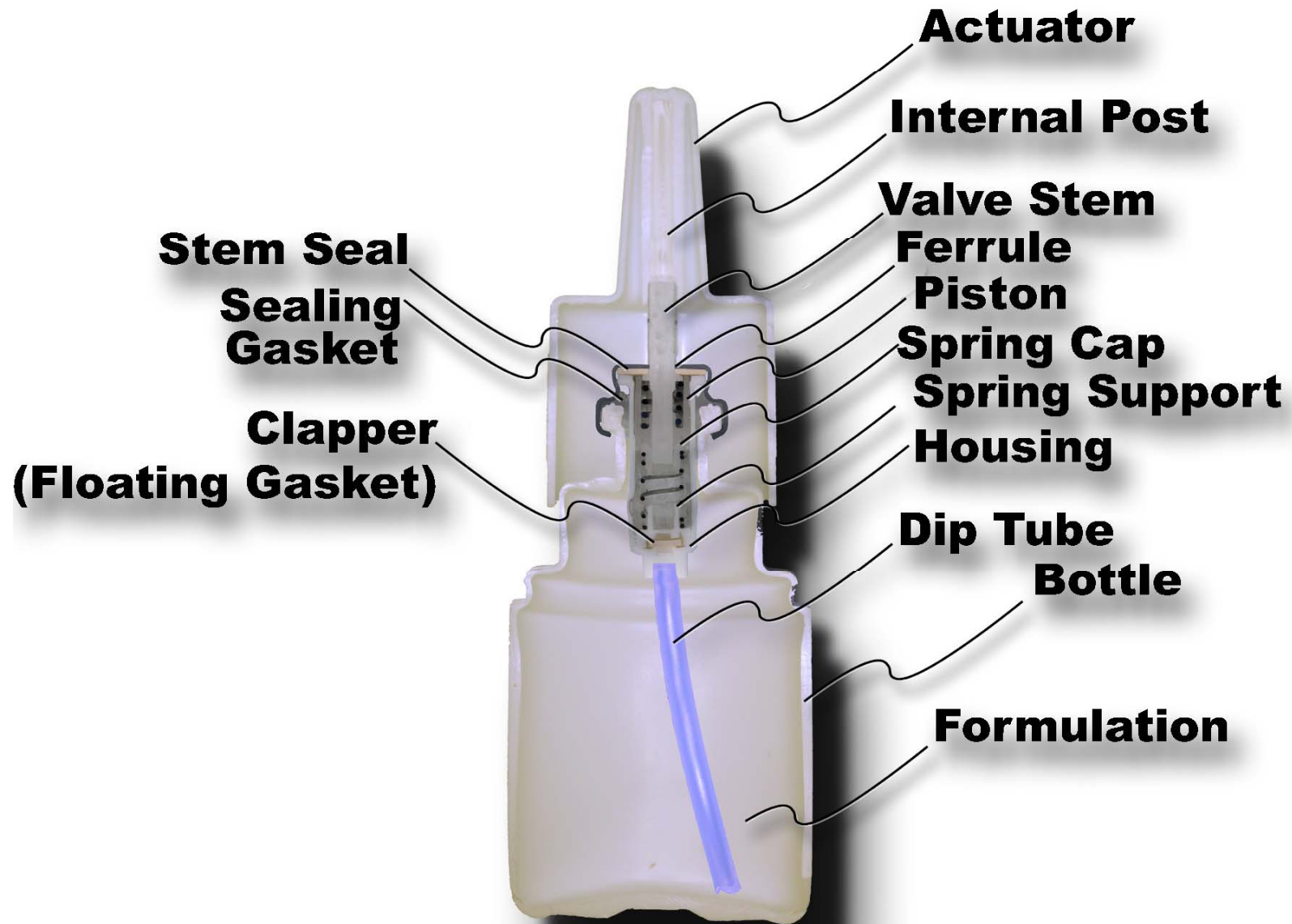
Pharmaceutical Delivery Systems

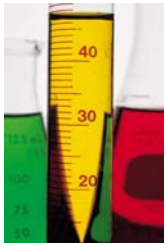
- **Plastics in medical devices are not currently regulated**
- **Unknown compounds may be added from raw material to finished product**
- **These additives can be detrimental to the product and to the consumer**



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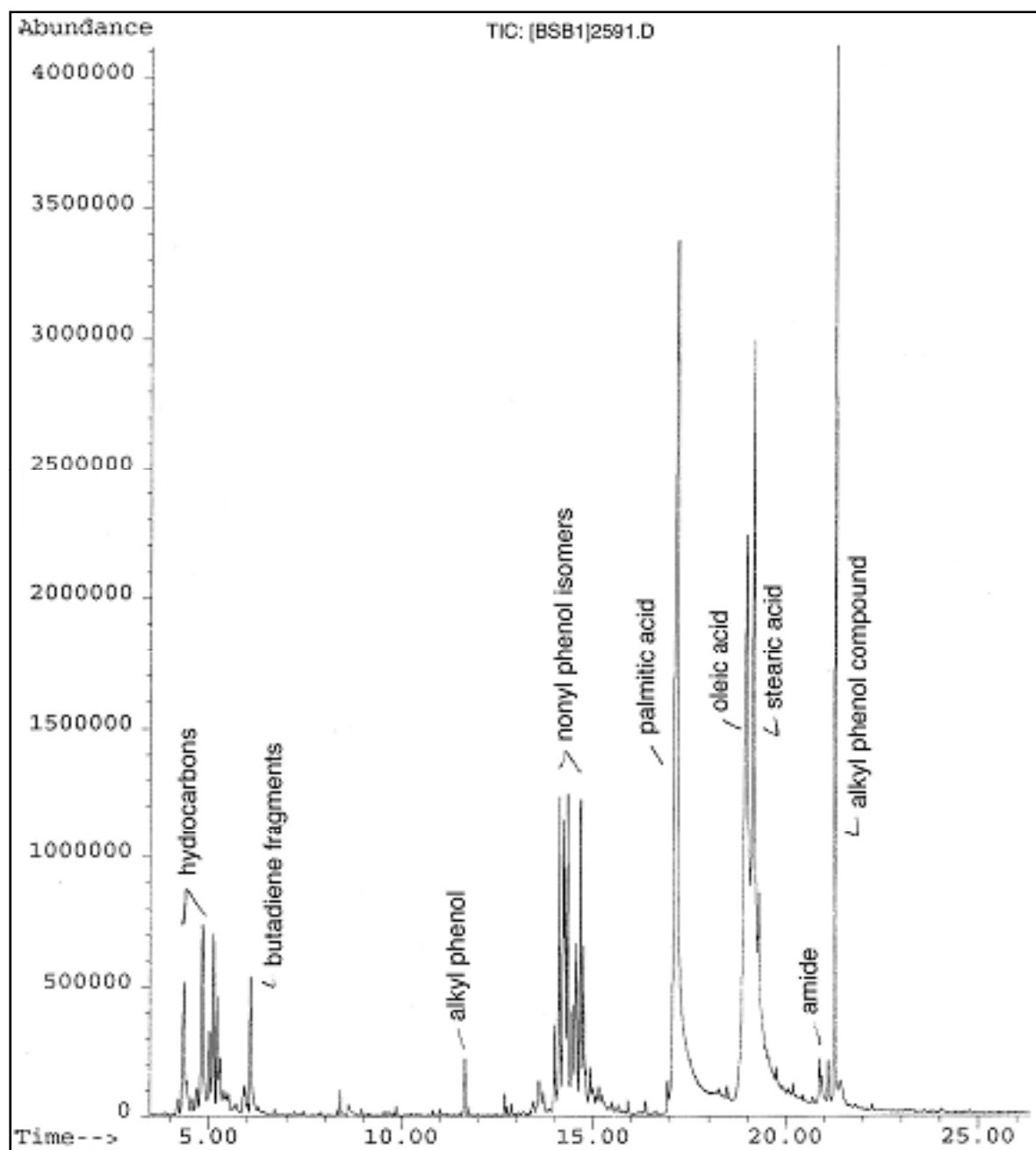


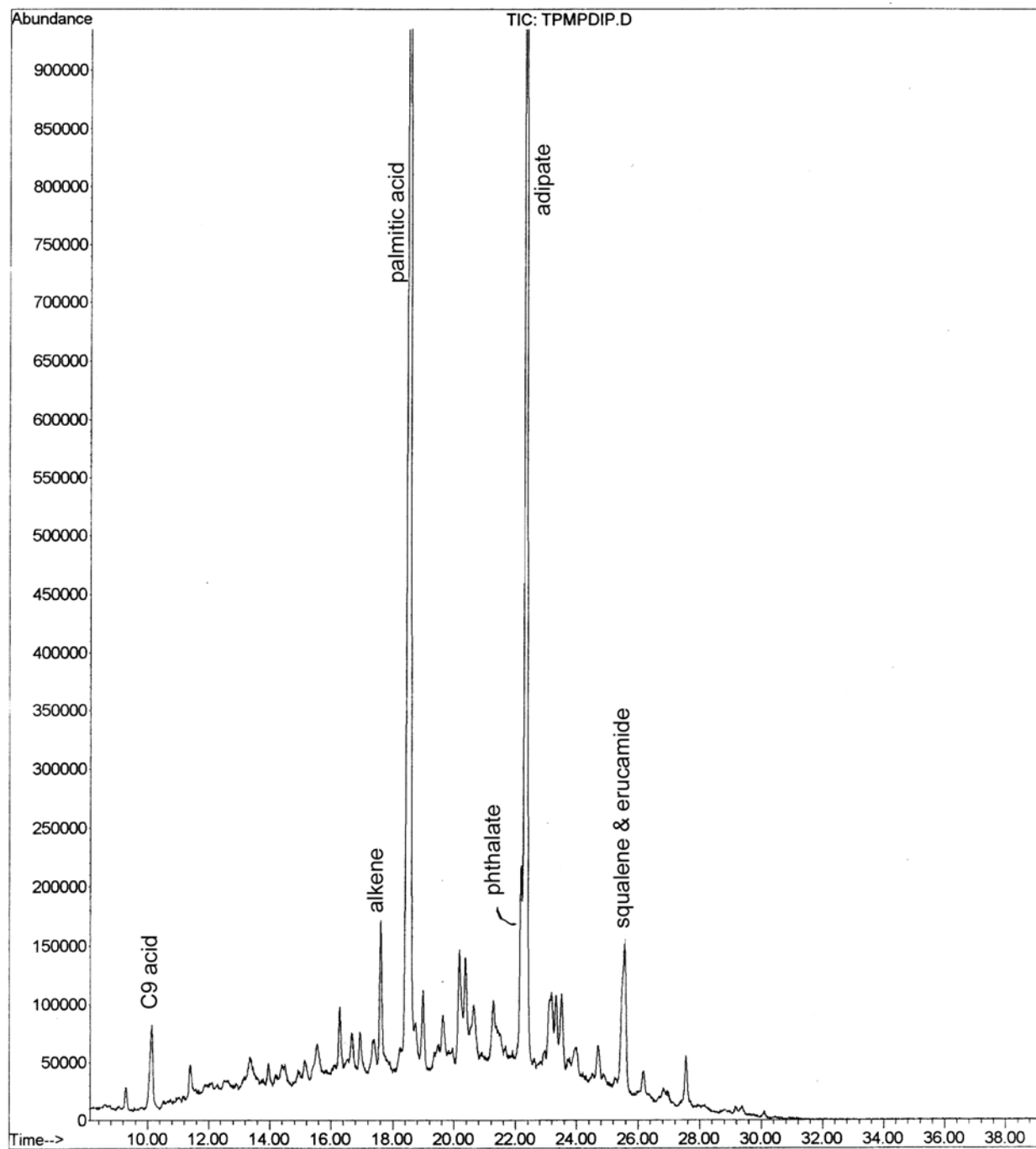
Gelled Product

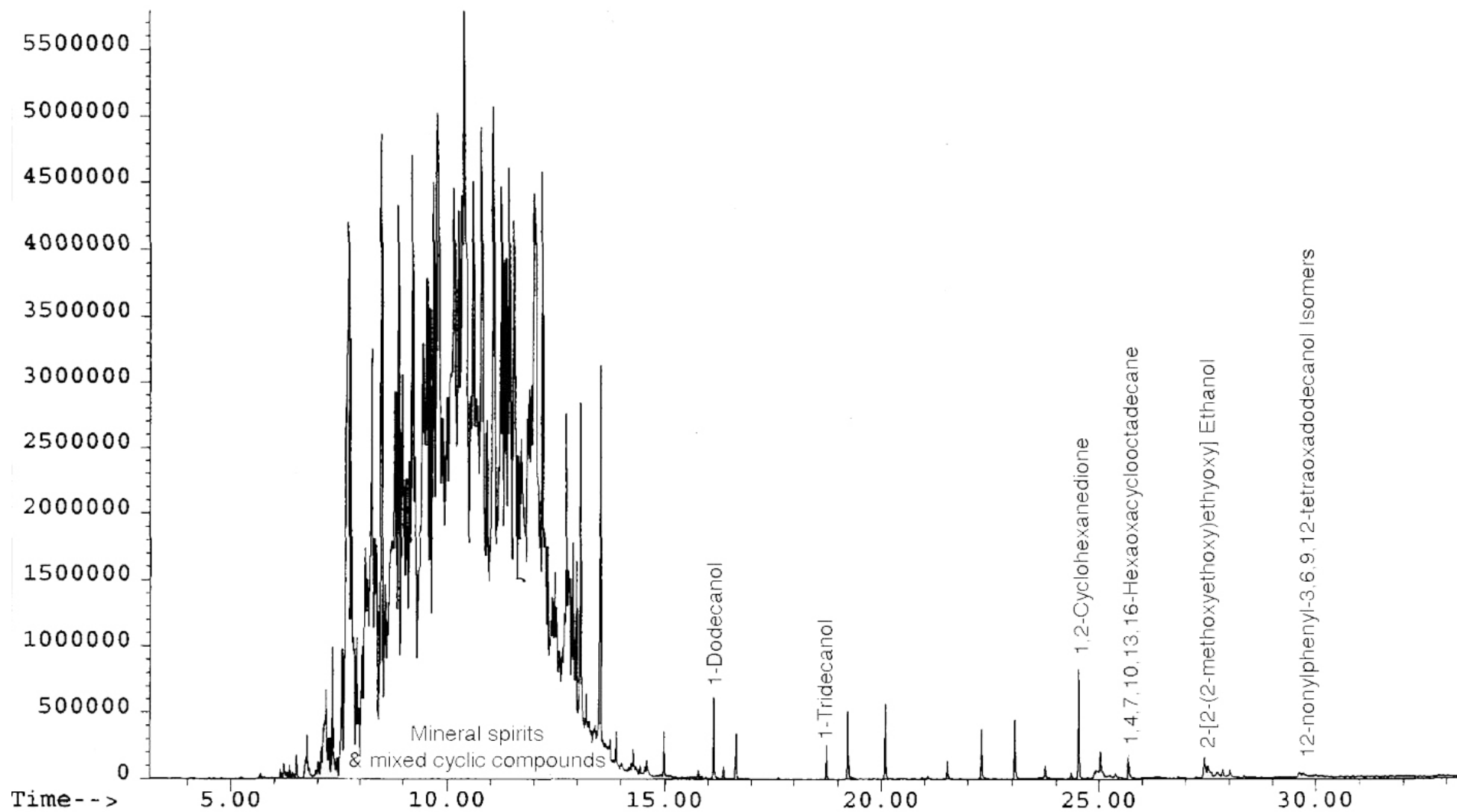


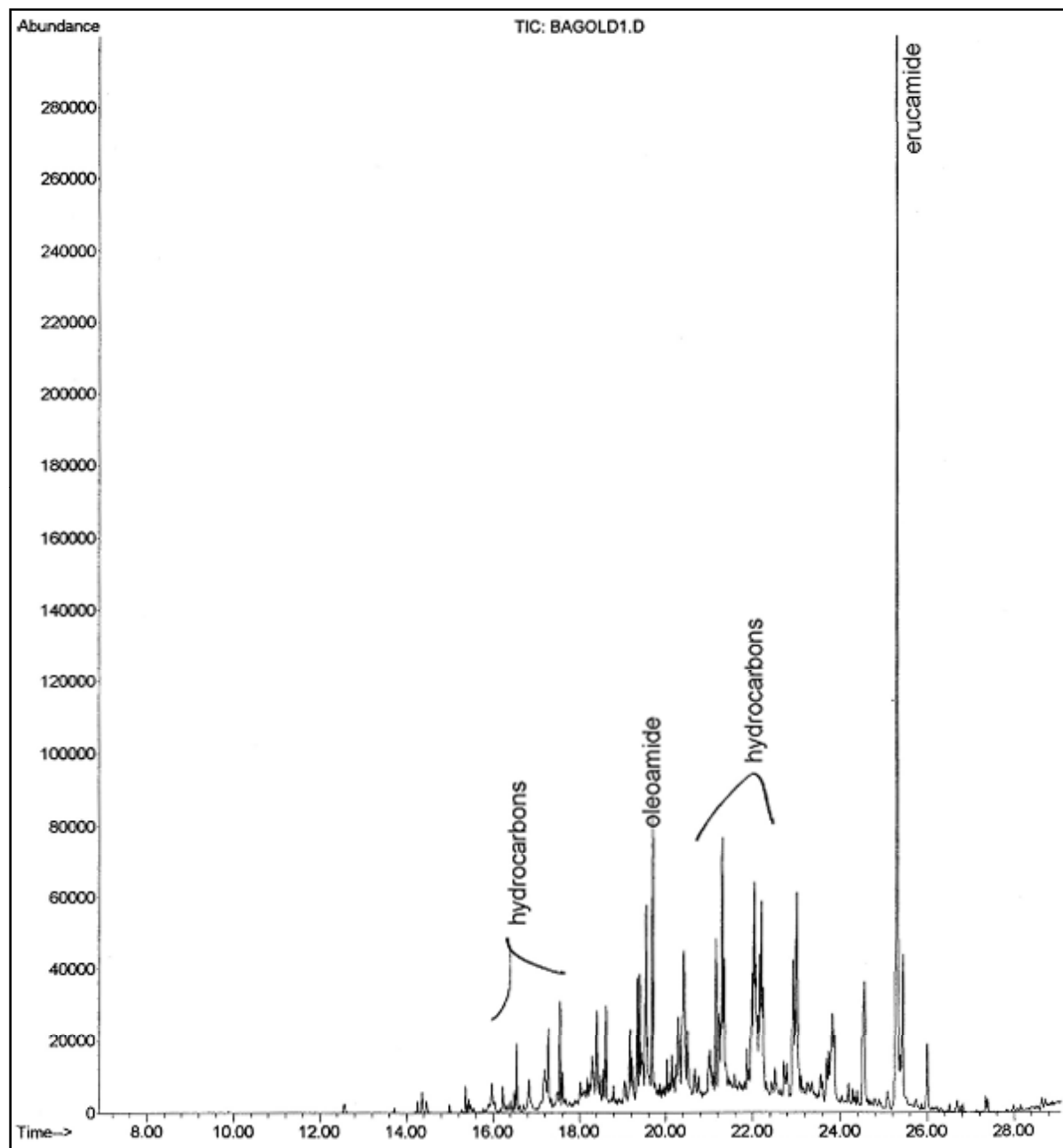
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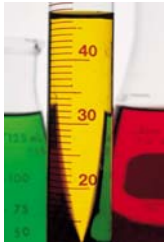
Conclusion

- A complete analysis of a polymer is possible with efficient extraction and a suitable analytical technique
- An array of properly characterized Chemical Reference Standards is required for the true determination of the additives contained a polymeric material



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Questions



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