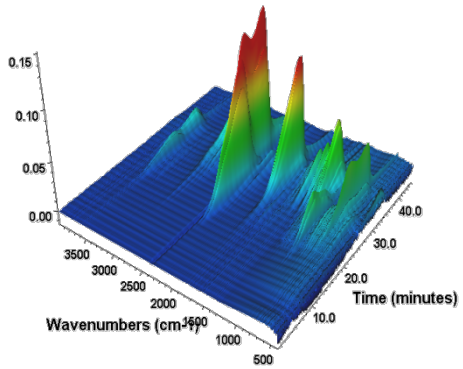
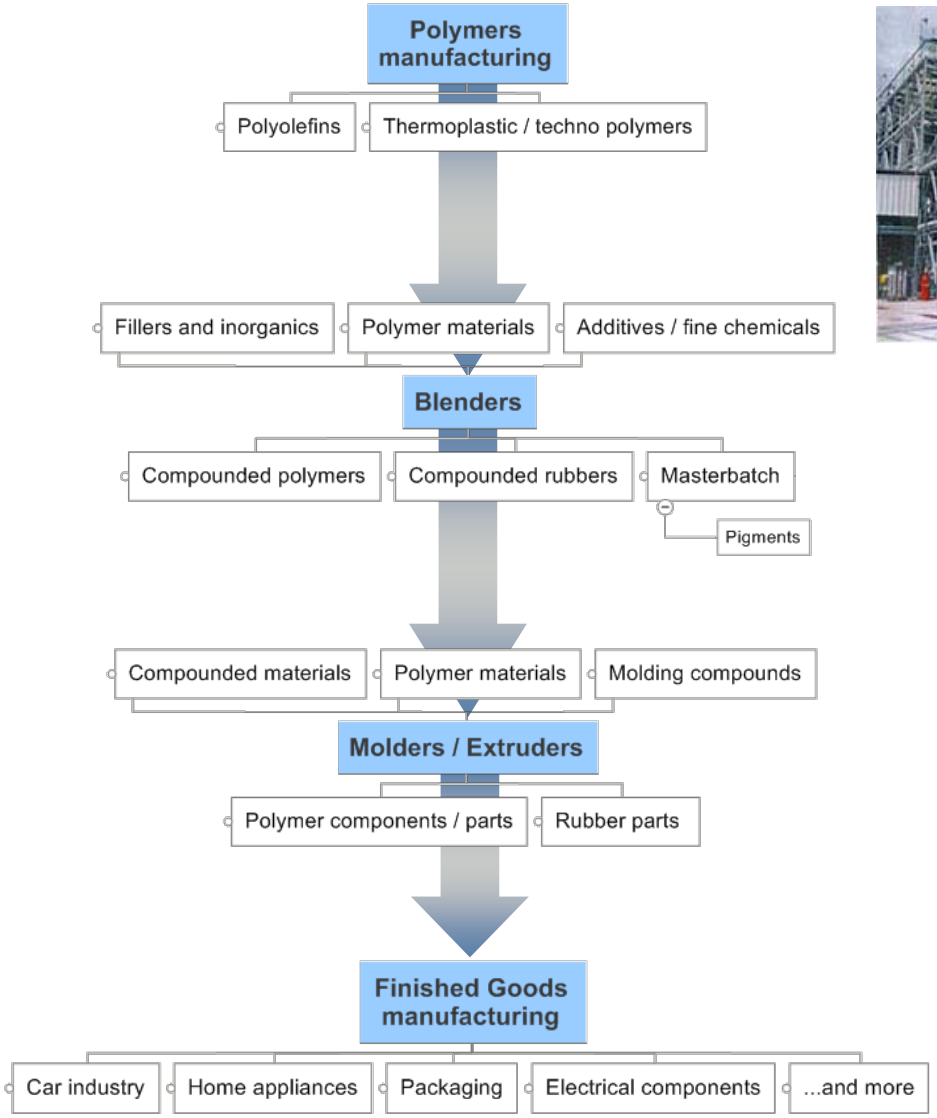
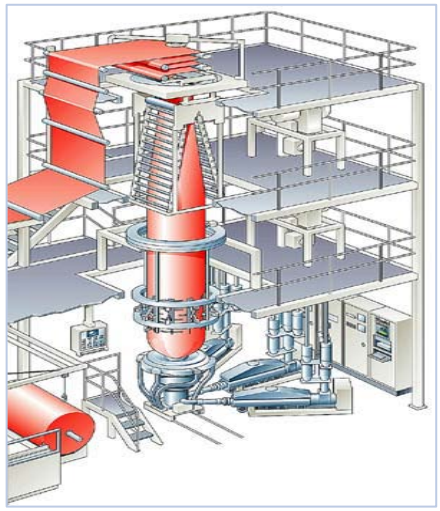


Breaking it Down

Polymer Deformulation Studies using FT-IR Coupled to TGA

Polymers Supply Chain





Molecular Spectroscopy in the Polymer Manufacturing Process

- **Material Characterization**
  - Control & Monitoring
- **Material Verification**
  - Incoming, In-process & Finished
- **Deformulation**
  - Root Cause Analysis & Reverse Engineering

Process NIR

NIR

Production

Receiving

Shipping

QC

R&D

Routine FT-IR

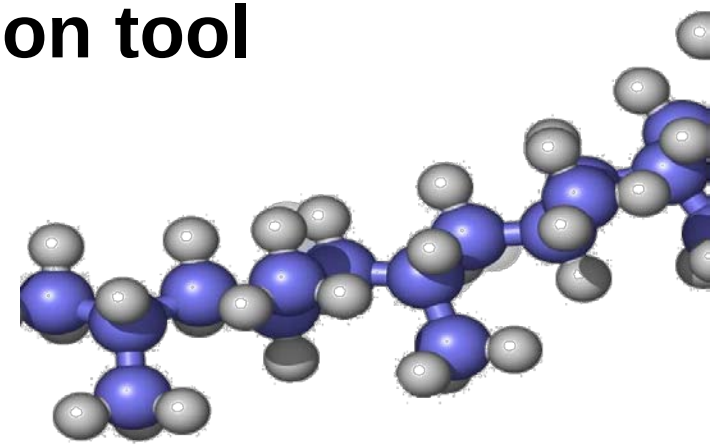
Advanced FT-IR

FT-IR Microscopy

Raman Microscopy

Overview

- **Deformulation**
  - What is it?
  - Why do it?
  - Common materials
- **FT-IR as a deformulation tool**
  - TGA coupled with FT-IR

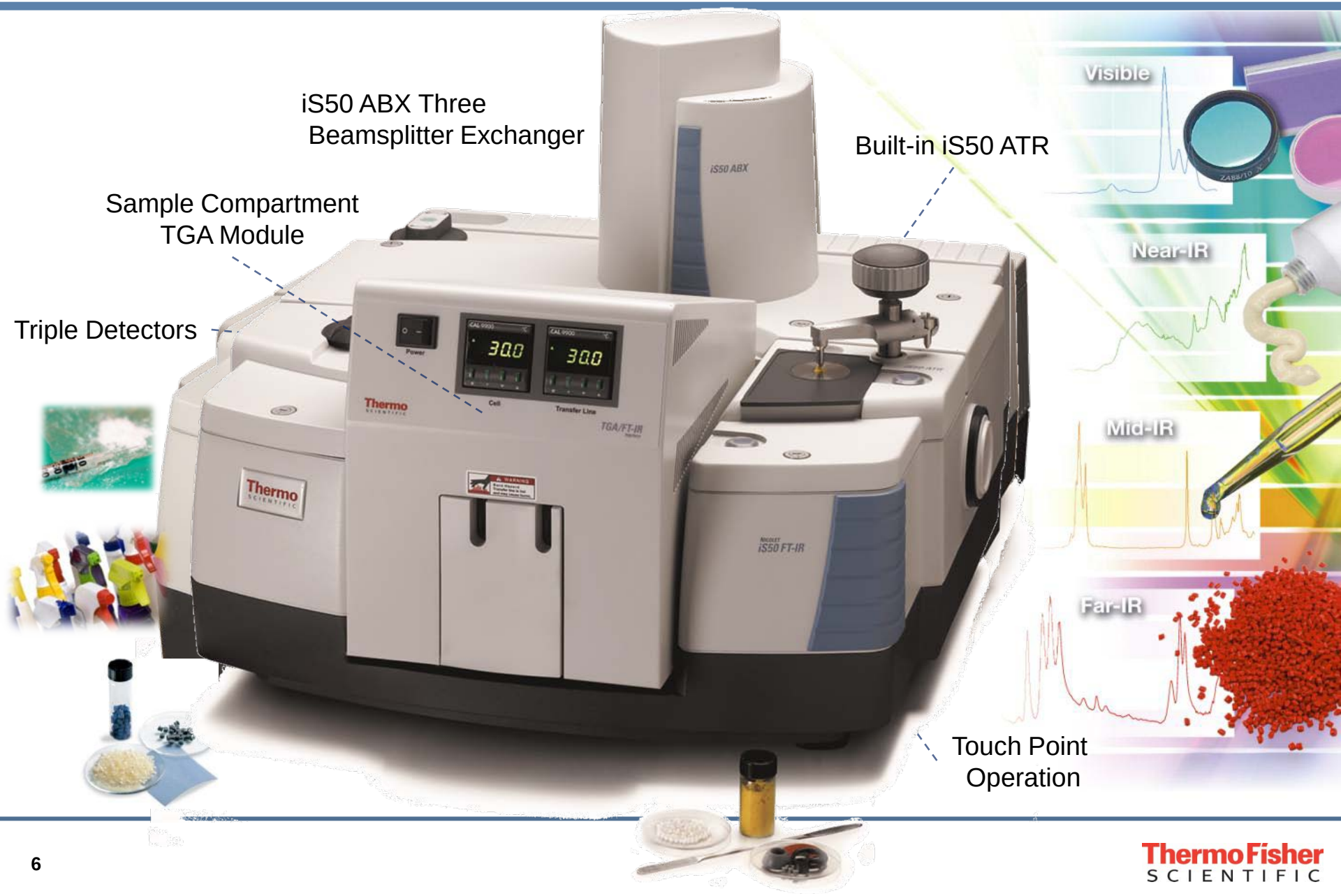


Deformulation

- Failure Analysis
  - Known material gone wrong
- Reverse Engineering
  - Unknown material composition

- Polymers
  - Plastics
    - Fillers, formulation
    - Biopolymers
  - Rubber
    - Carbon Black: O-Rings, Tires
- Epoxies, Resins, Adhesives
- Entrained solvents, breakdown products
- Fabrics, Paper products

The Nicolet iS50 FT-IR



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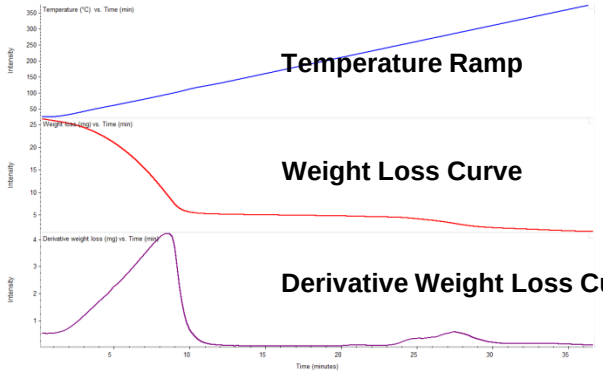
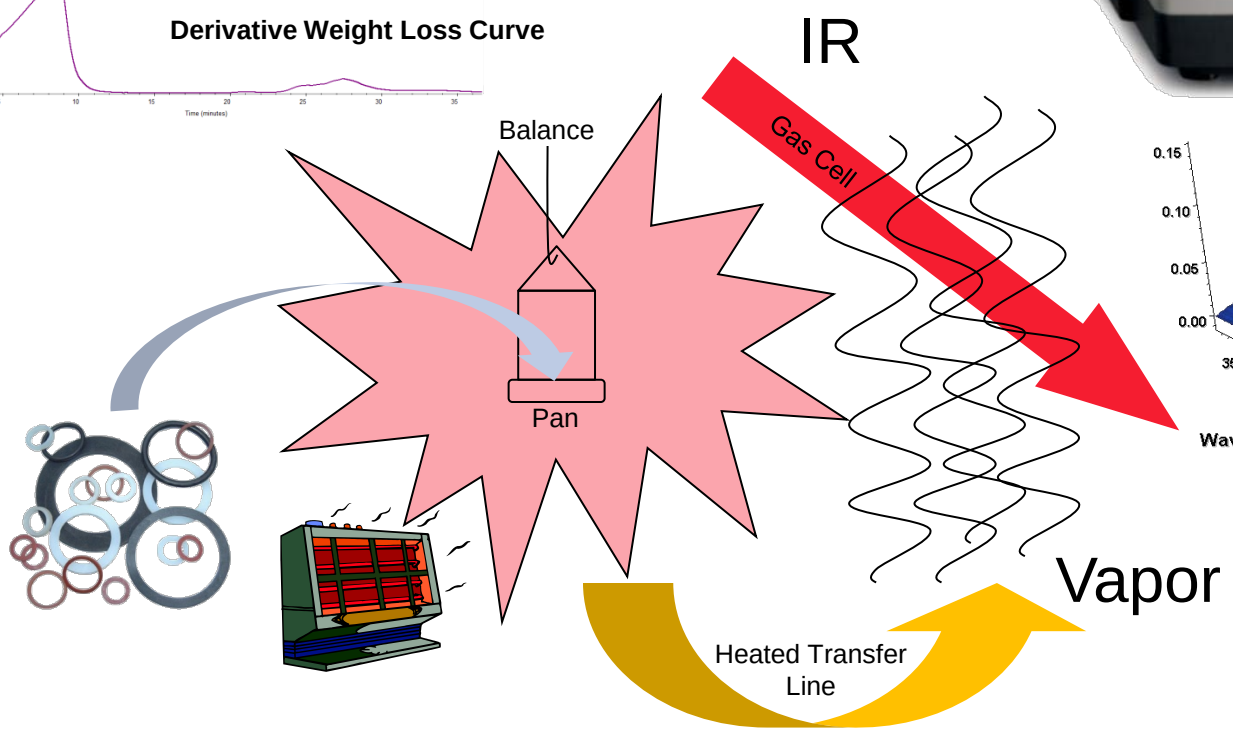
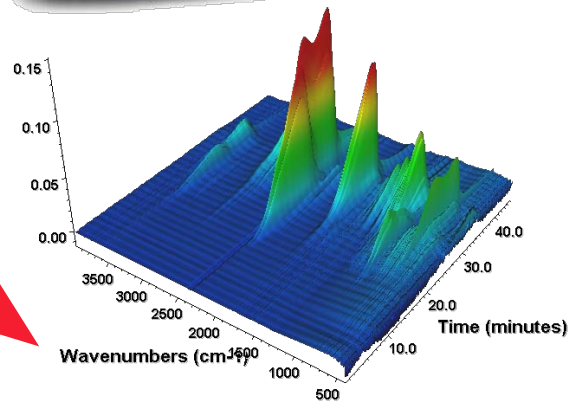
TGA-IR: The Basics







Infrared Series File

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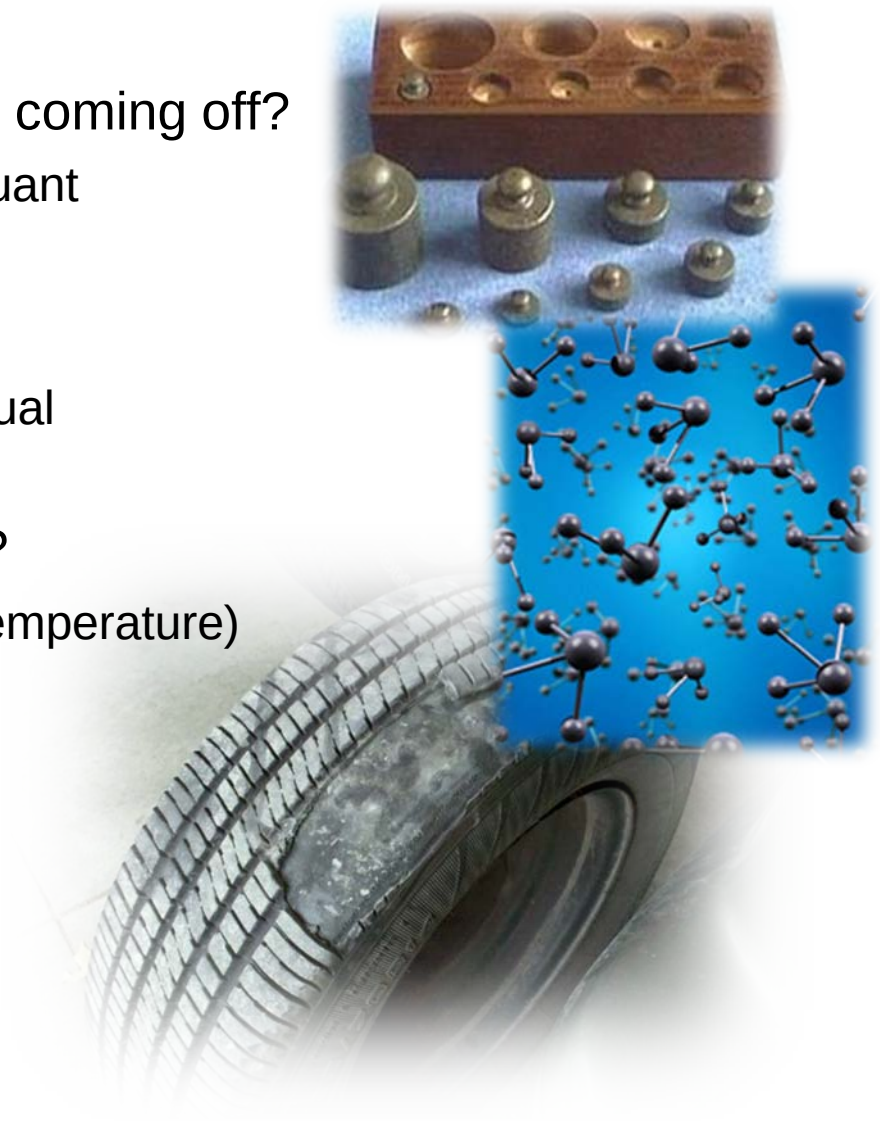
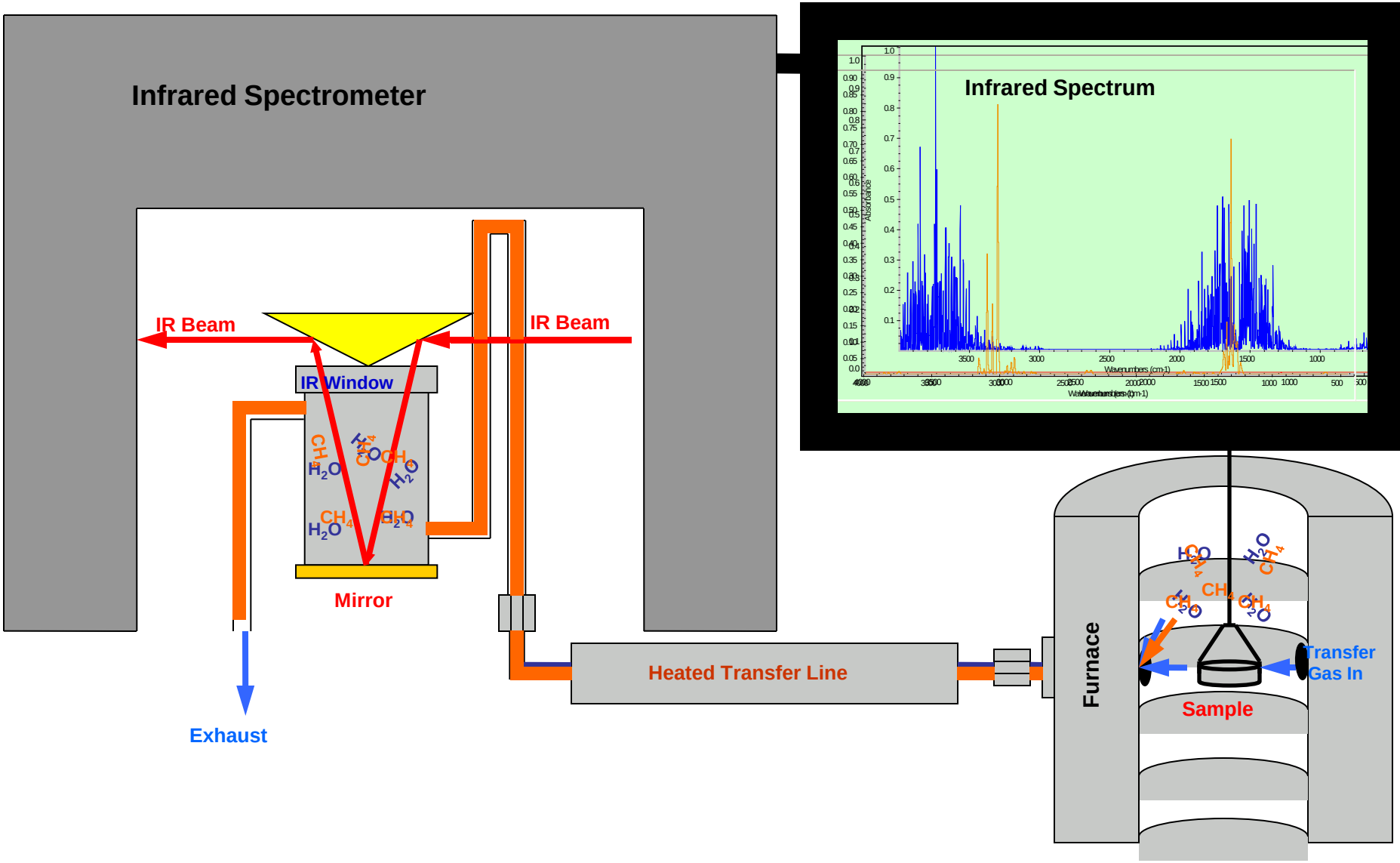
Desired Information

- Quantitative: How much material is coming off?
  - T “G” A – Gravimetric Analysis = quant
- Qualitative: What came off?
  - FT-IR – Vibrational information = qual
- Process: Similar yet different, why?
  - Profiles of materials versus time (temperature)
- It takes all three to get formulation



TGA-FTIR: Experiment



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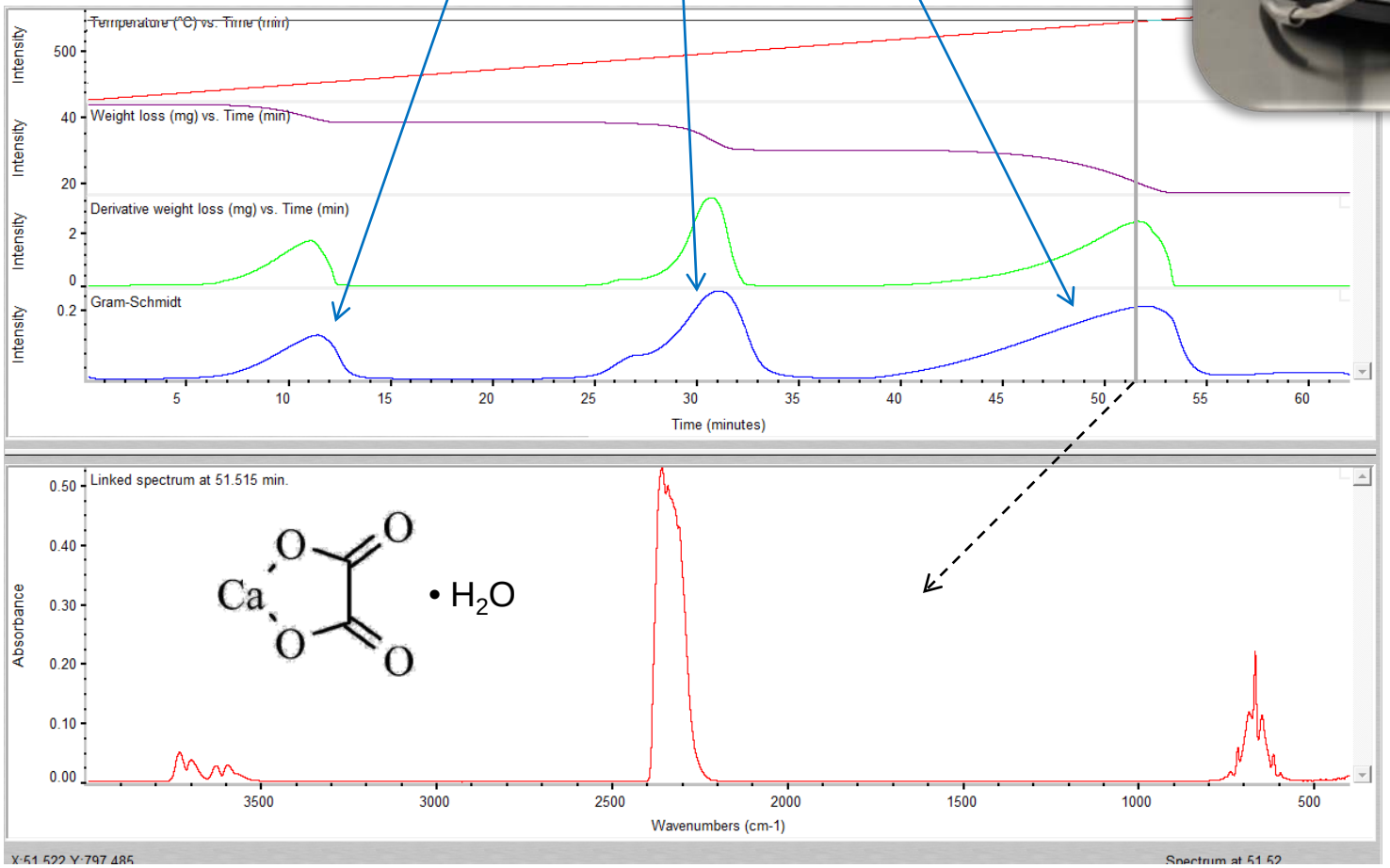
Simple Situation – Calcium Oxalate

- Three weight-loss peaks
  - H_2O ; $CO + CO_2$; CO_2



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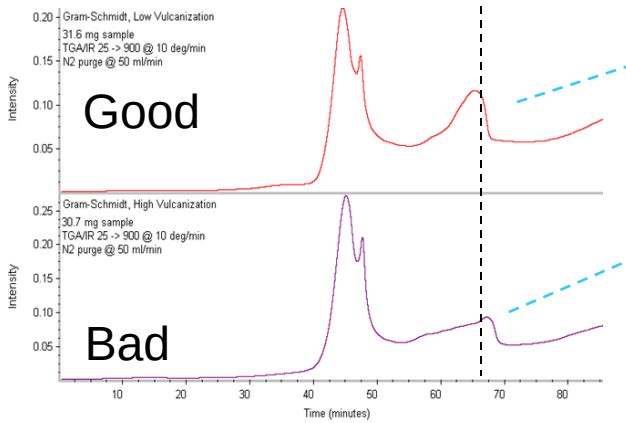
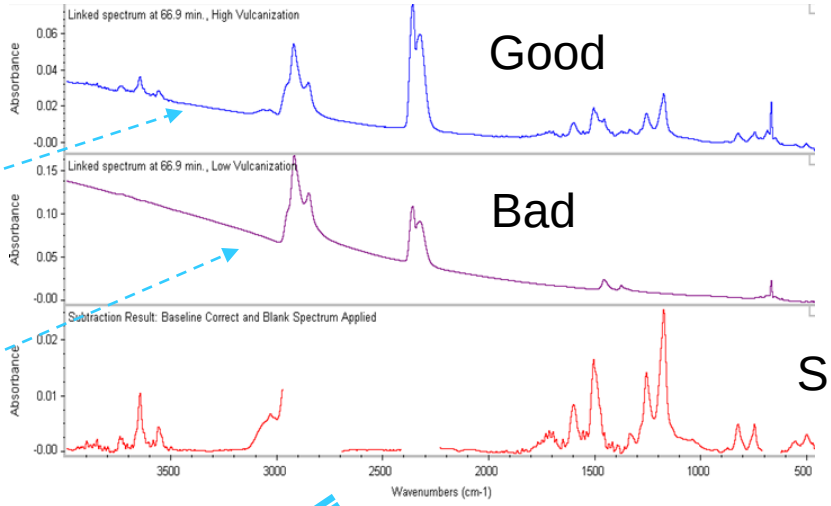
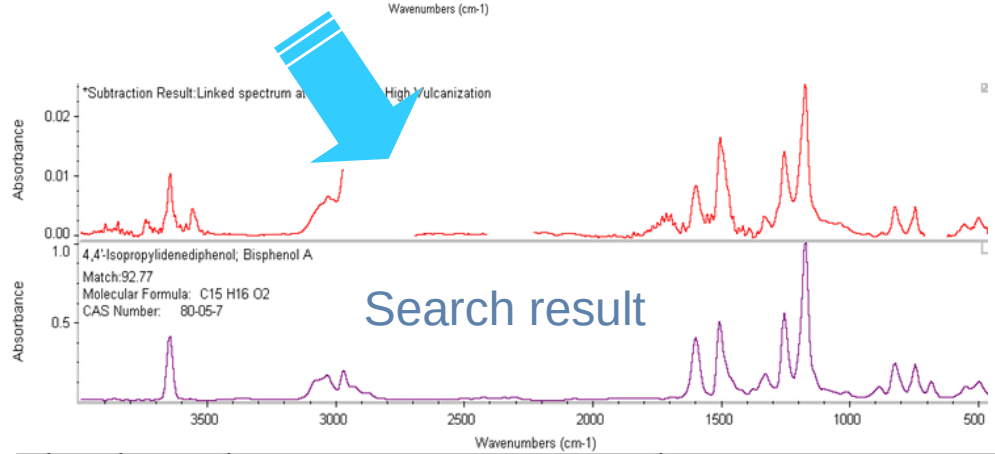
Classic Root Cause Analysis

Gaskets analysis

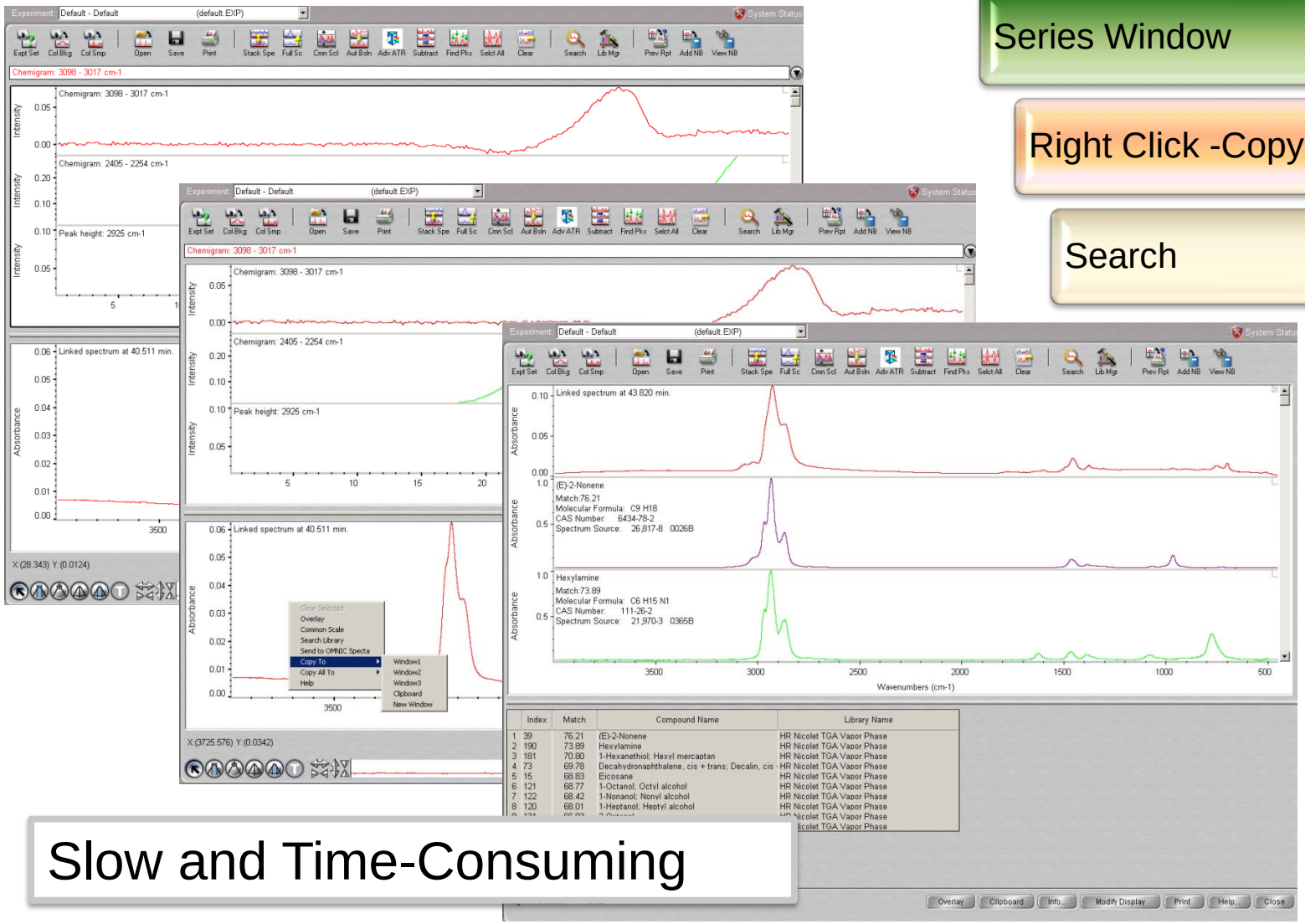






	Index	Match	Compound Name	Library Name
1	3269	92.77	4,4'-Isopropylidenediphenol; Bisphenol A	Nicolet/Aldrich Vapor Phase
2	28	87.20	4,4'-Isopropylidenediphenol; Bisphenol A	Hazardous Chemicals - Vapor Phase Spectra
3	5307	87.17	4,4'-Isopropylidenediphenol; Bisphenol A	Nicolet Vapor Phase
4	407	87.17	4,4'-Isopropylidenediphenol; Bisphenol A	Nicolet TGA Vapor Phase
5	5306	85.83	4-(2-Phenylisopropyl)phenol; 4-Cumylphenol	Nicolet Vapor Phase

TGA Current State



Series Window

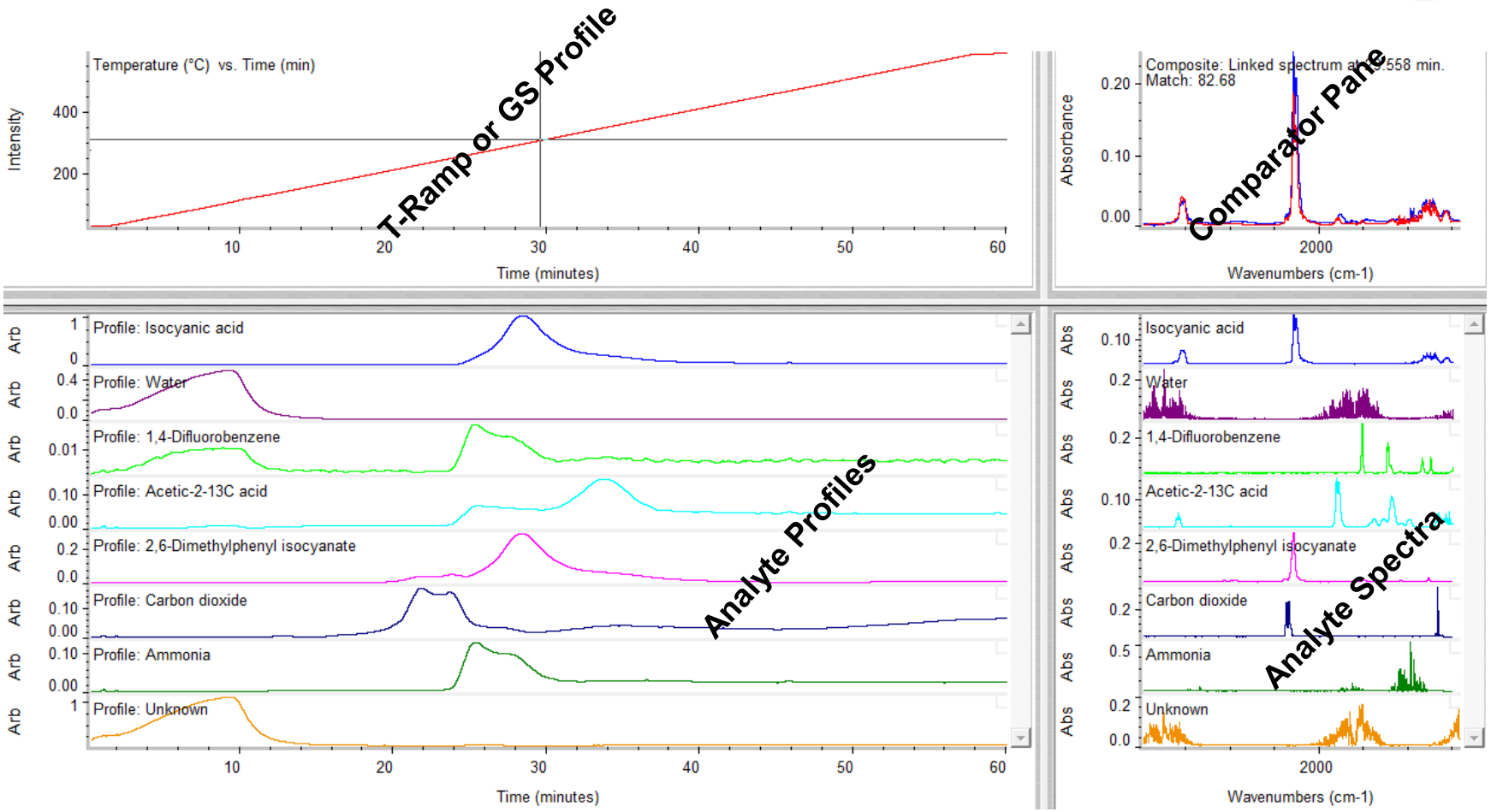
Right Click -Copy

Search

Slow and Time-Consuming

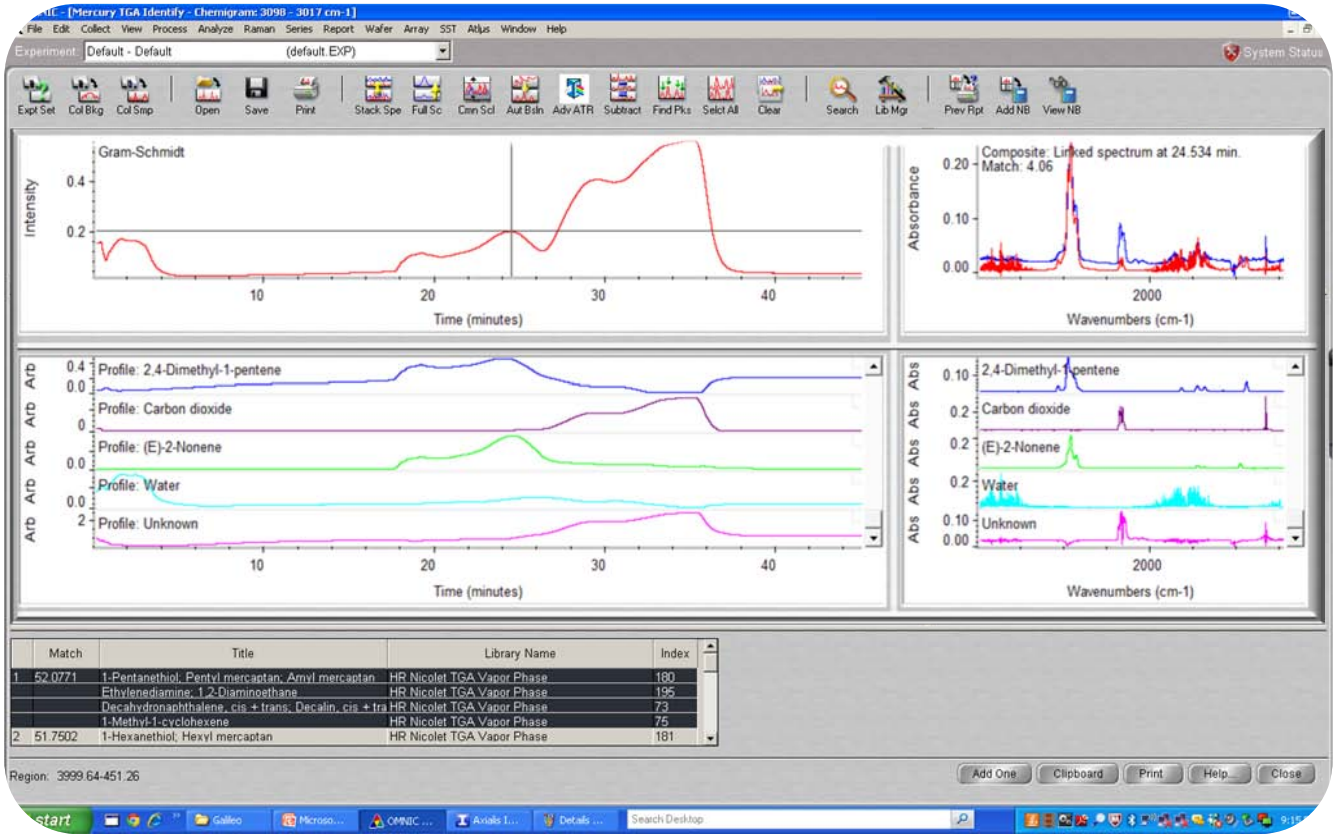
ThermoFisher SCIENTIFIC

Complete Analysis: Mercury TGA



ThermoFisher SCIENTIFIC

Mercury TGA



Fast Answers with Search Results, Component Profile Information and Match information all in one window!

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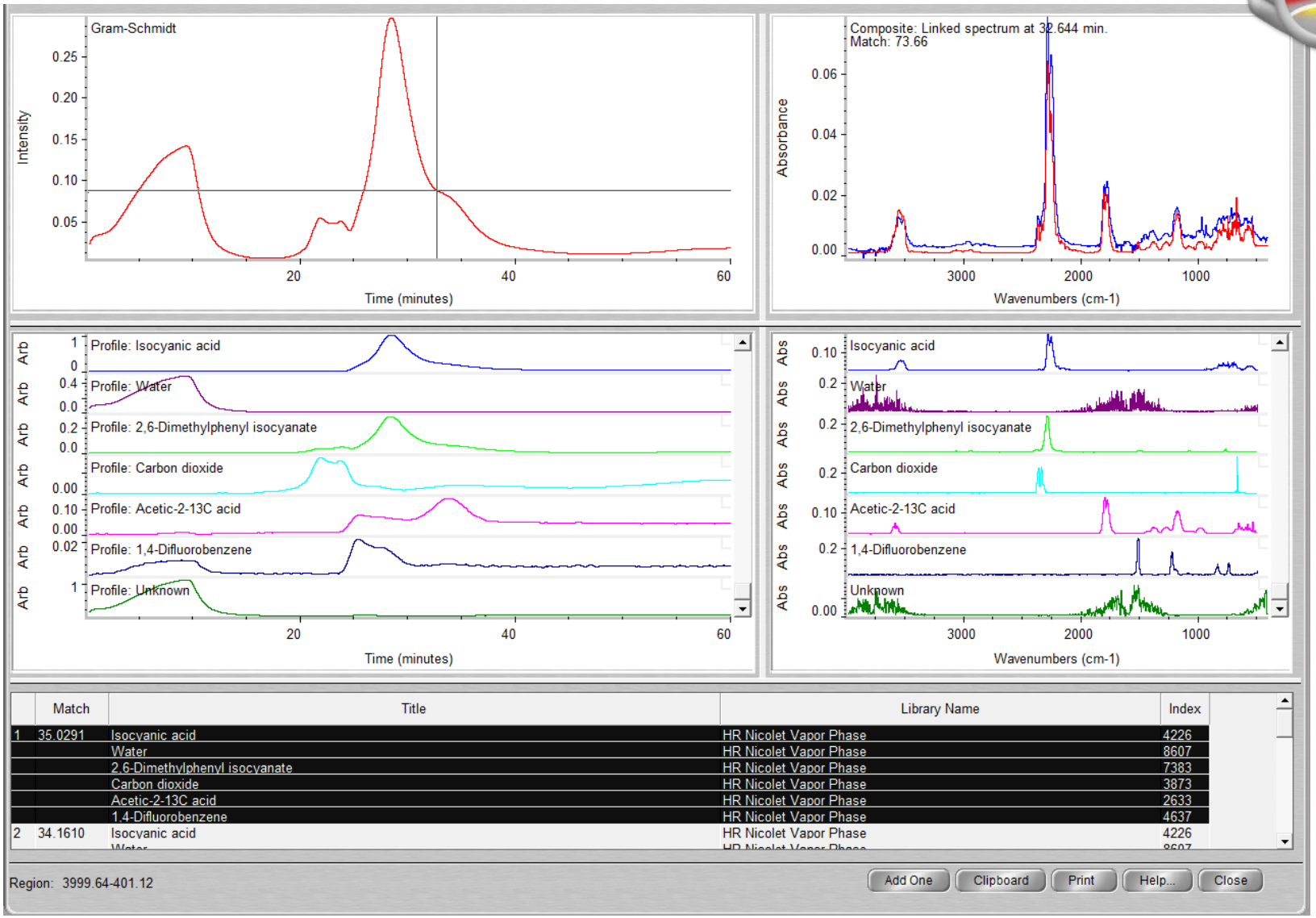
How Many Components?



	Match	Title	Library Name	Index
1	35.0291	Isocyanic acid	HR Nicolet Vapor Phase	4226
		Water	HR Nicolet Vapor Phase	8607
		2,6-Dimethylphenyl isocyanate	HR Nicolet Vapor Phase	7383
		Carbon dioxide	HR Nicolet Vapor Phase	3873
		Acetic-2-13C acid	HR Nicolet Vapor Phase	2633
		1,4-Difluorobenzene	HR Nicolet Vapor Phase	4637
2	34.1610	Isocyanic acid	HR Nicolet Vapor Phase	4226

Region: 3999.64-401.12

Add One | Clipboard | Print | Help... | Close

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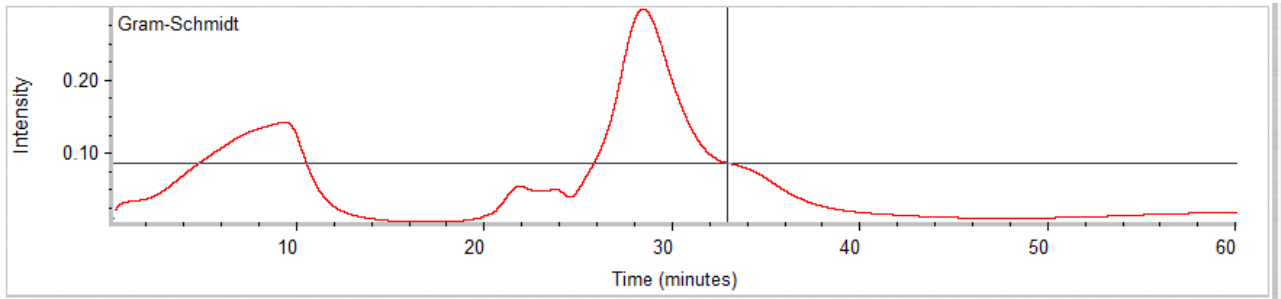
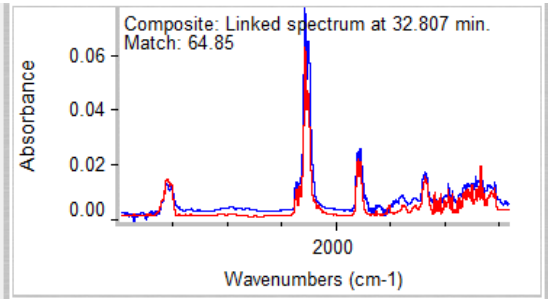
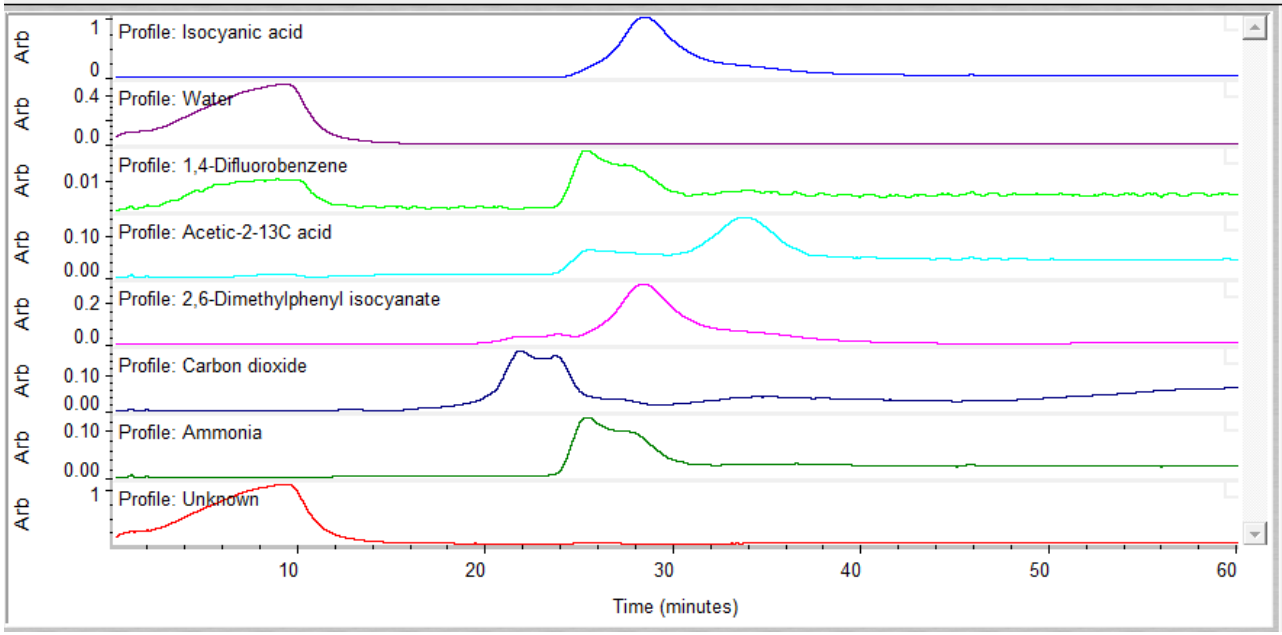
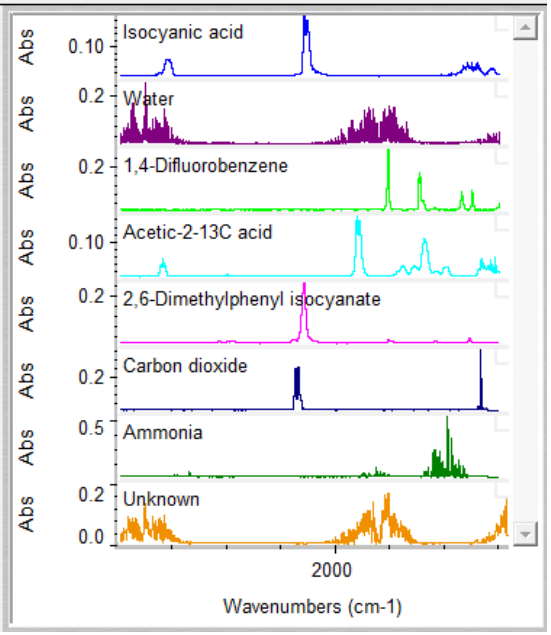
Full Deformulation: 7 Components









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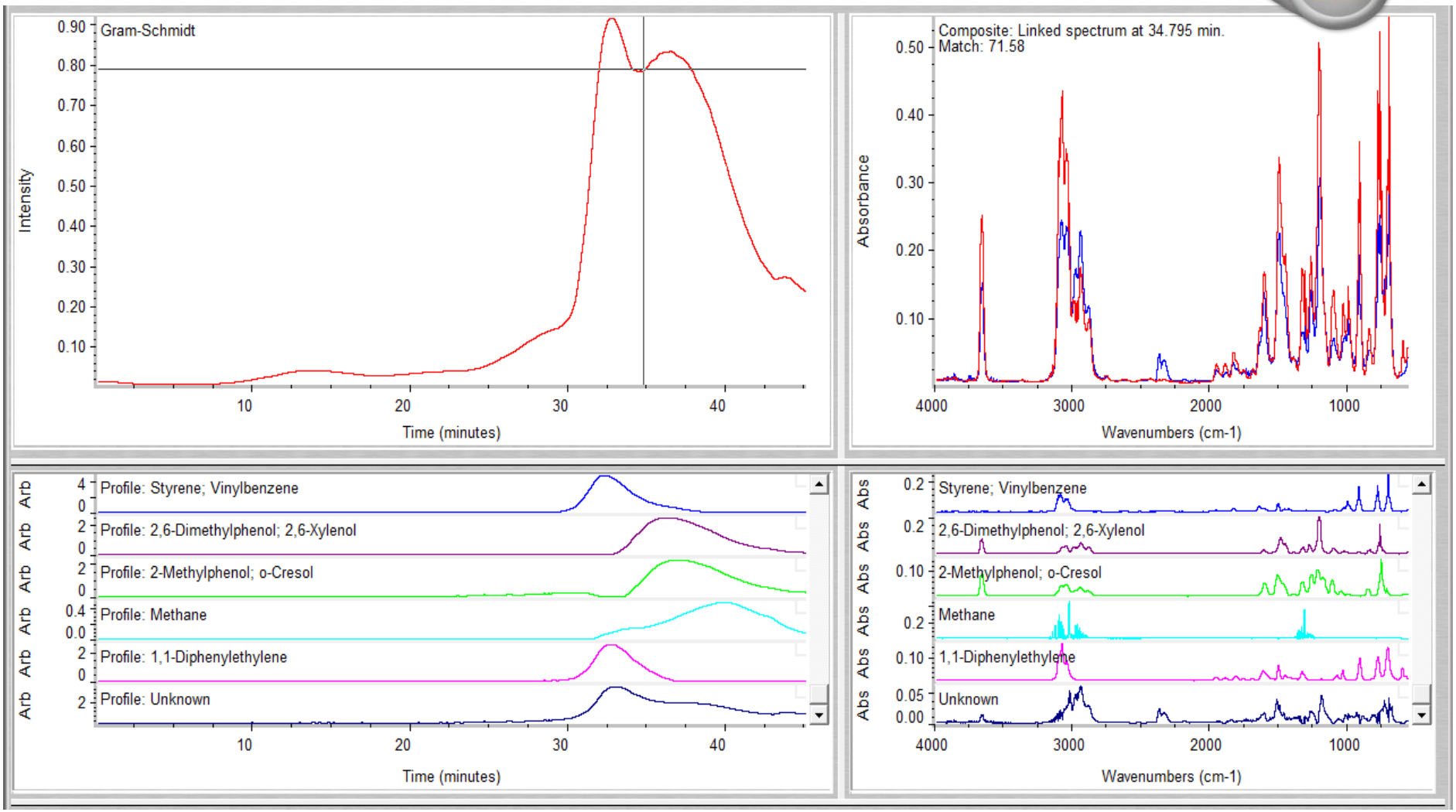
Structural Foam



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TGA-IR Analysis of Museum Materials

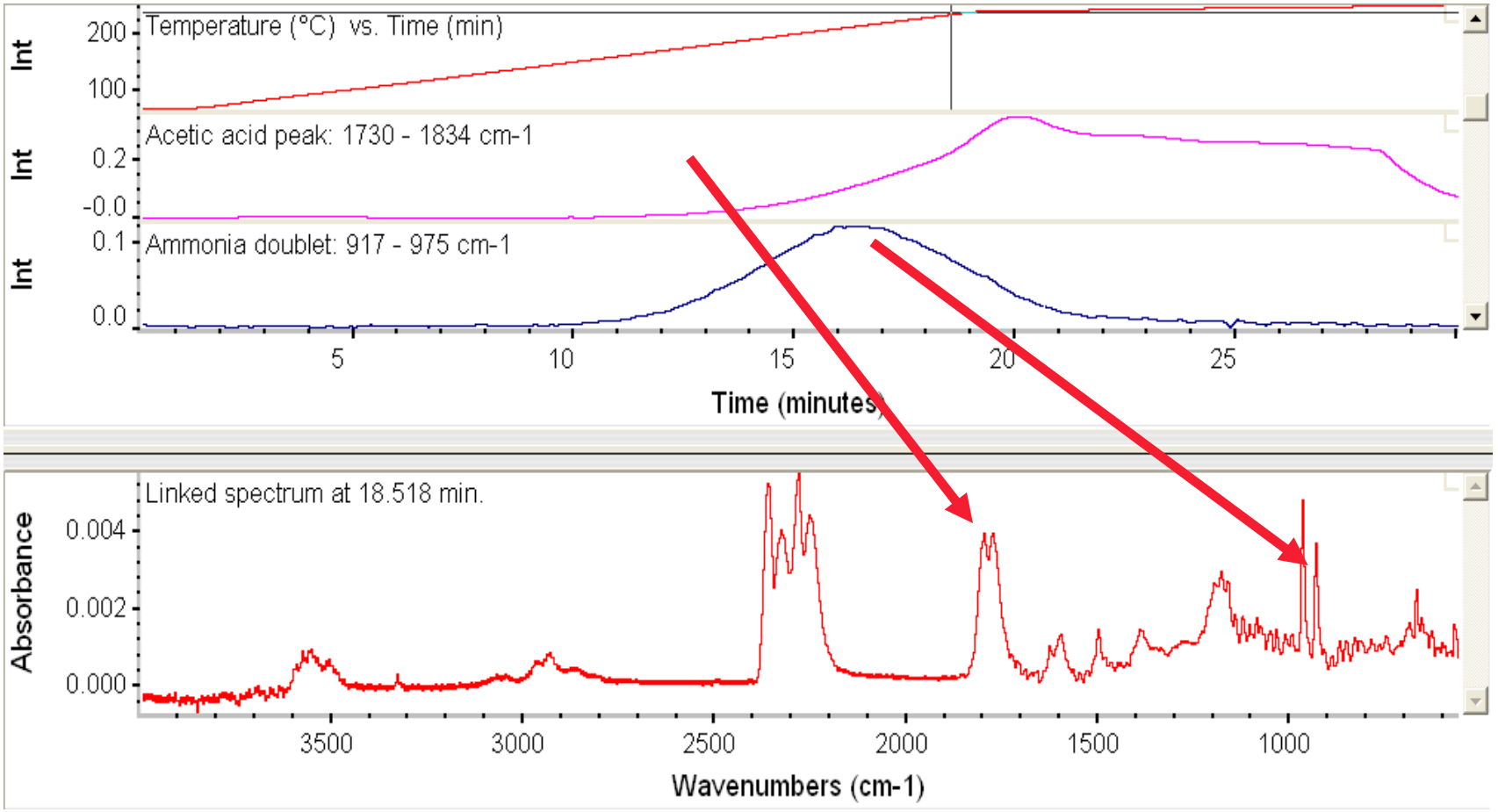
- Museum needs to display artifacts
- Must balance preservation with aesthetics
- What if display material slowly outgases?
  - Low level concentrations
  - Encased material – concentrates gases
  - Long term exposure possible

Mummy Case of Paankhenamun

Cartonnage (gum, linen, and papyrus) with gold leaf, Third Intermediate Period, Dynasty 22, c. 945-715 B.C.; h. 170.2 cm; William M. Willner Fund, 1910.238.

Art Institute of Chicago

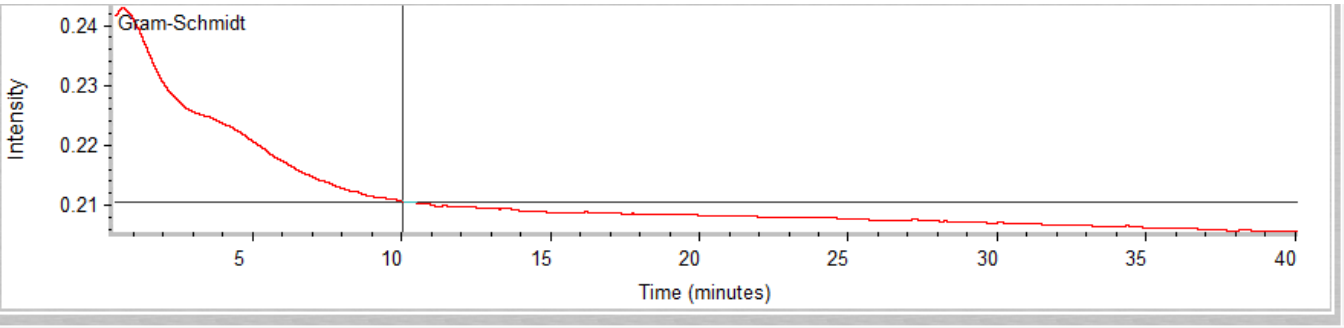
TGA-IR Data of Display Materials: Velvet

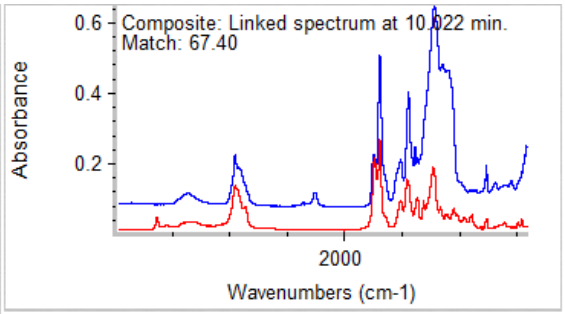


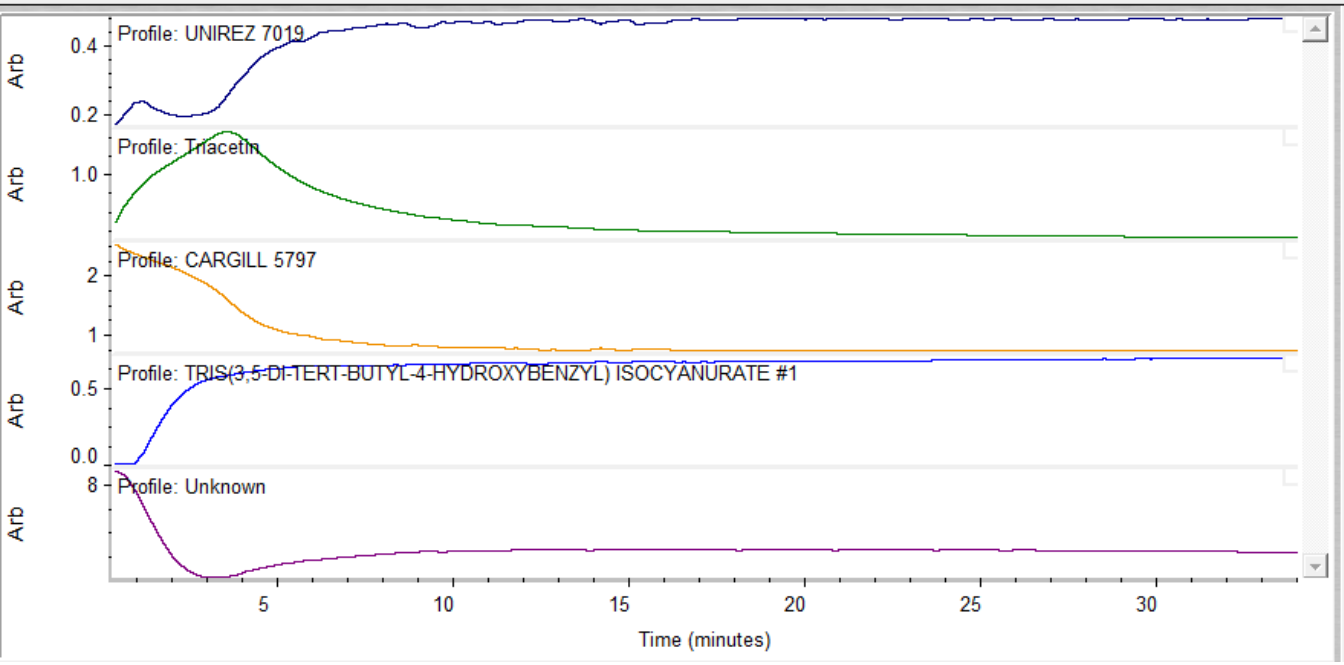
Ammonia and acetic acid detected

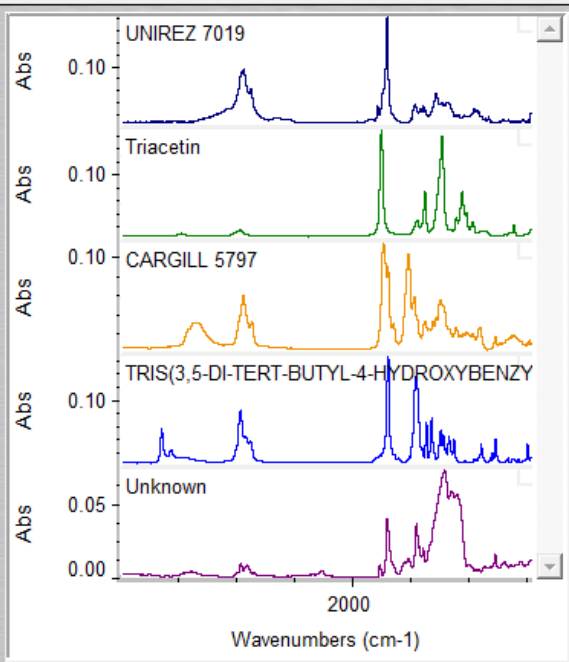
Mercury TGA Also Works for Kinetics!

- Reaction of urethane









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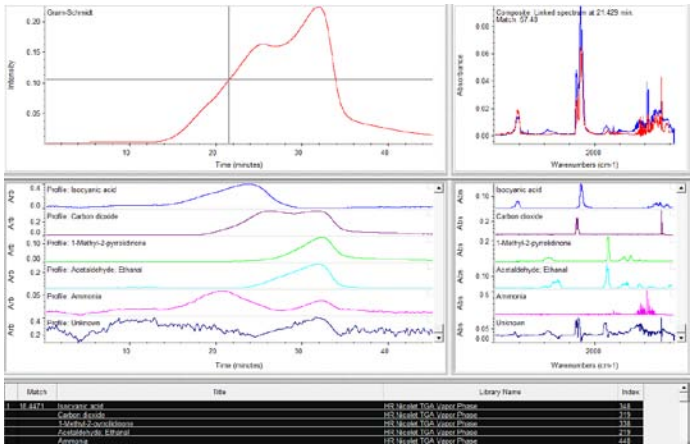
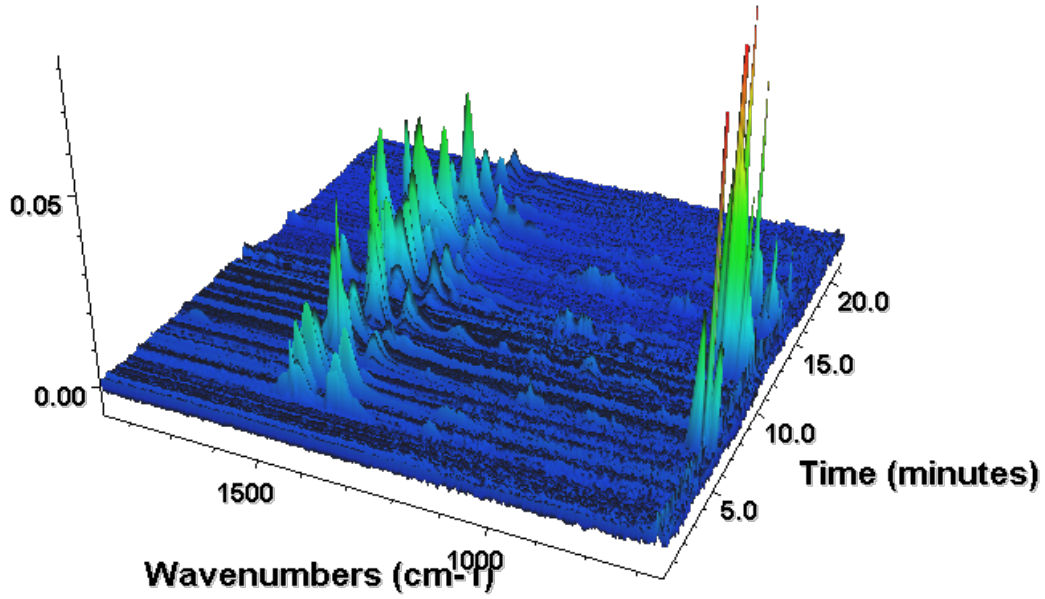
Conclusions - Deformulation using FT-IR & TGA

- TGA coupled to FT-IR advances capabilities to break materials down
- Mercury TGA software simplifies the analysis of complex mixtures





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mike.garry@thermofisher.com

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