Uncover the insight with Hyphenation

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PerkinElmer®
For the Better

Let’s start from the beginning

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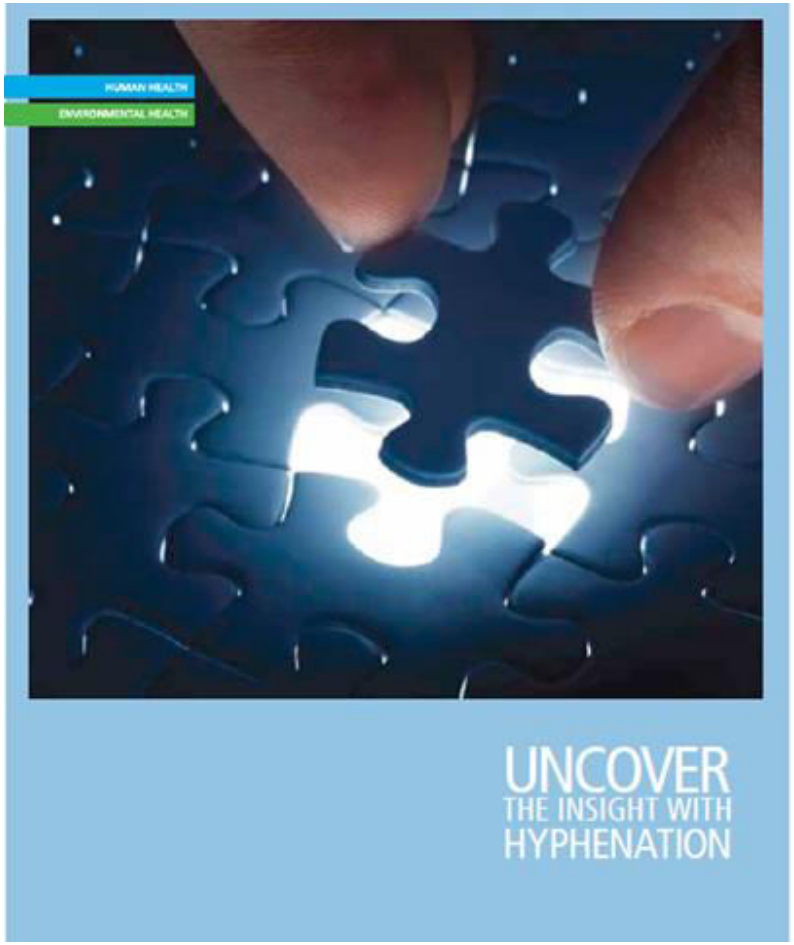
Outline

- Material Characterization at PerkinElmer
- Thermal Analysis
  - DSC, TMA, DMA, TGA
    - And some examples
- Hyphenation
  - What is hyphenation?
  - Evolving Gas Analysis
    - TGA-IR
    - TGA-MS
    - TGA-GCMS
    - TGA-IR-GCMS
  - And some of their applications



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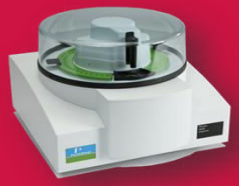
Material Characterisation at PerkinElmer

UV/Vis /NIR Spectroscopy

- From routine QA/QC to high demanding UV/Vis/NIR applications
- Academia, Pharma, Coatings, Glass ....

IR Spectroscopy

- From simple Identification for incoming materials to research grade FTIR/FTNIR applications
- Polymer, Pharma, Academia, Food ....



Thermal Analysis

- To measure thermal properties for QA/QC and process optimization
- Polymer, Pharma, Academia ....

UV/Vis/NIR product portfolio

Performance

Lambda HighPerformance UV/Vis NIR
Lambda 650/750/850/950/1050

PerkinElmer UV software
UV Winlab for HPUV
UV Express for Lambda 365
UV Lab for Lambda 265/465

Lambda 465

UV/Vis/NIR-Spectrometer
High performance across the UV/Vis/NIR range
Unmatched flexibility and sensitivity

Lambda 365

UV/Vis-Spectrometer
High performance PDA that delivers reliability – and confidence

Lambda 265

UV/Vis-Spectrometer
compact, versatile high-performance double-beam UV/Vis

PDA UV/Vis-Spectrometer
fast, accurate, affordable results

Lambda XLS/Bio

UV/Vis-Spectrometer – stand alone
easy, fast, robust and reliable

..the broadest product portfolio - for each application the right solution

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IR product portfolio

Performance

Spectrum 10 and Touch™ Software
ONE software package
for the complete FTIR product line

Spotlight Microscopy and Imaging
for Frontier and Spectrum Two

Frontier MIR/NIR/FIR systems

Problem solving/R&D

FT-IR for highest demand

IR FOR
EVERYBODY
EVERYWHERE
EVERYDAY

QA/QC
mobile
robust
routine

Spectrum Two

Solution Systems:
Lubricants
Biofuels
Environmental
Polymers
Pharma
Education
Nutraceuticals &
TCM
…

IR READY
TO GO

IR READY
FOR ANY
CHALLENGE

..the broadest product portfolio - for each application the right solution

Thermal analysis product portfolio

DSC 8000/8500
Double furnance DSC (power compensation)

PERKINELMER PYRIS

PerkinElmer Pyris Software
A single software platform for all

TGA 8000
For high demand

designed for Hyphenation

DMA 8000

TMA 4000

STA 6000/8000
Simultanoues TG/DTA

Reliability at it's best DSC
for routine & QA/QC
applications

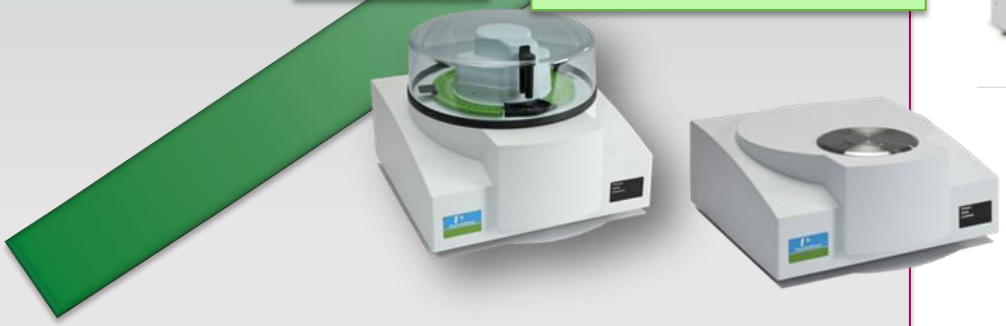
DSC 4000/6000
Single furnance DSC

TGA 4000

TGA for routine & QA/QC
applications

Innovation in
Materialscience
TMA & DMA for various
applications

Performance



..the broadest product portfolio - for each application the right solution

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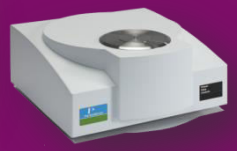
Thermal Analysis - Techniques

DSC 8000, 8500

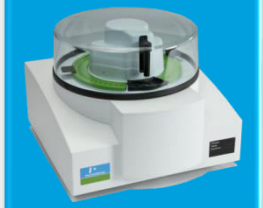


DSC 4000, 6000

Glass Transition, Melting/Crystallization, Reaction Rate, Specific Heat, Curing, Crosslinking

DMA 8000

Modul, Expansion, Damping, Alpha and Beta Transition, Glass Transition, Melting



STA 6000, 8000

TGA 4000

TGA 8000

Wt % additives & loss of additives, Wt % filler & ash, Decarboxylierung, Pyrolyse, Decomposition and Stability studies

Hype-nation

Identification Quantification of evolved gases in Polymers and its Components using FTIR, MS or GCMS

The complete PerkinElmer Productportfolio in Thermal Analysis

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Thermal Analysis

	DSC	TGA	DMA	TMA
Full name	Differential Scanning Calorimetry	Thermogravimetric Analysis	Dynamic Mechanical Analysis	Thermomechanical Analysis
Property	Enthalpy	Weight change	Elasticity	Dimensions
Glass transition	✓		✓ ✓	✓ ✓
Melting	✓ ✓		✓	✓
Crystallization	✓ ✓		✓	✓
Specific heat capacity	✓			
Thermal history	✓ ✓		✓	✓
Curing, polymerization	✓ ✓	✓	✓	✓
Evaporation, dehydration	✓	✓		
Thermal decomposition	✓	✓ ✓		
Modulus, stiffness			✓	✓
Thermal expansion/shrinkage				✓

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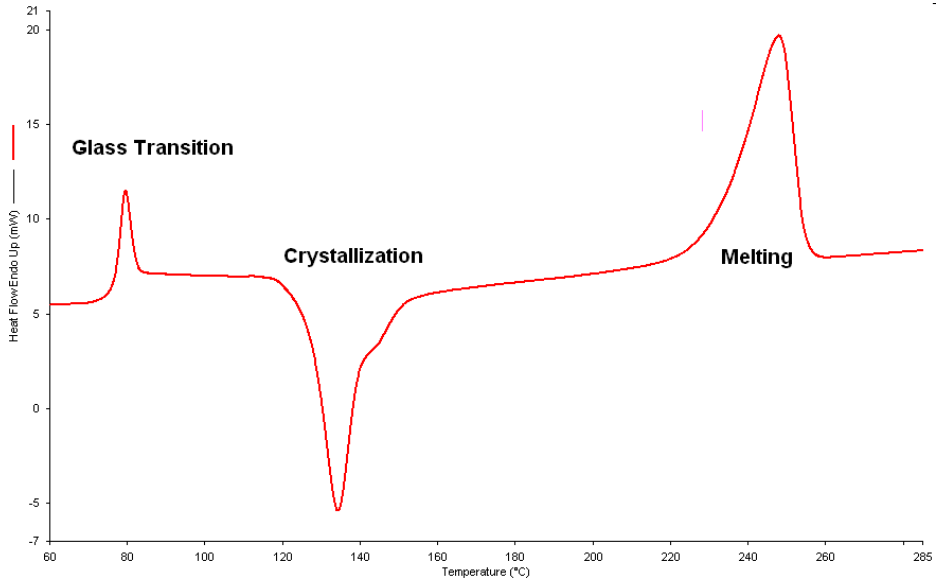
DSC – Differential Scanning Calorimeter
What does DSC measure?

- DSC measures the amount of energy (heat) absorbed or released by a sample as it is heated, cooled or held at constant temperature. DSC also performs precise temperature measurements.

DSC is used to analyze

- Melting
- Crystallization
- Glass Transition
- O.I.T. (Oxidative Induction Time)
- Polymorphism
- Purity
- Specific Heat
- Kinetic Studies
- Curing Reactions
- Denaturation



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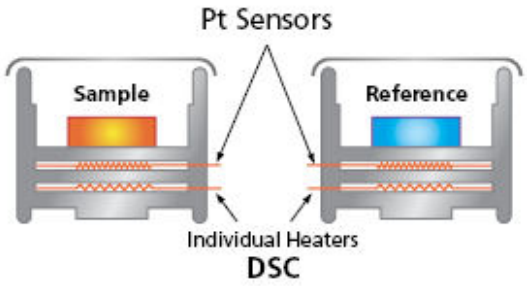
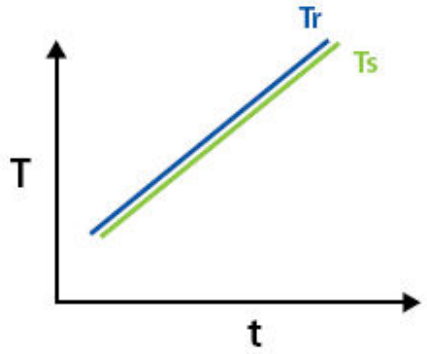
DSC – Differential Scanning Calorimeter
Power Compensation vs Heat Flux

Power Compensation Double-furnace DSC

Directly measures heat flow between two independent, low-mass furnaces.

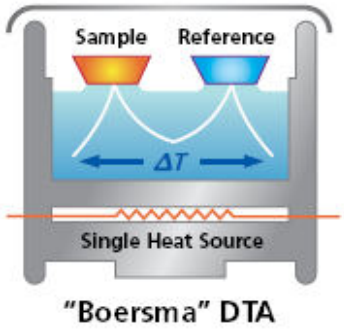


- Dual independent small furnaces
- Null principle, ΔT=0, measures ΔH directly/accurately

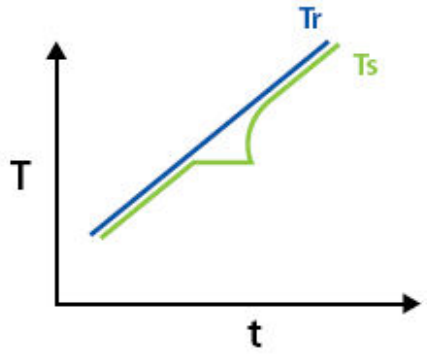


Heat Flux Single-furnace DSC

Measures temperature differential between sample side and reference side using single, large-mass furnace.
Needs mathematical equations to determine the heat flow.



- Single-furnace design
- Drives ΔH from ΔT signal



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DSC – Differential Scanning Calorimeter
Mass and volume comparison

- Main reasons why we can do fast scan DSC
  - Smaller furnaces
    - More >100g (heat flux) versus <3g (power compensated)
    - Better temperature controlled on a small thermal mass
  - 90% Pt furnace
    - With its highly conductive material, the Pt furnaces are much easier to heat up and cool down
  - The furnace cradles the sample
    - The sample heats faster and more evenly
  - Powerful heater
    - Cover the whole bottom of each furnace to give an even and maximum power

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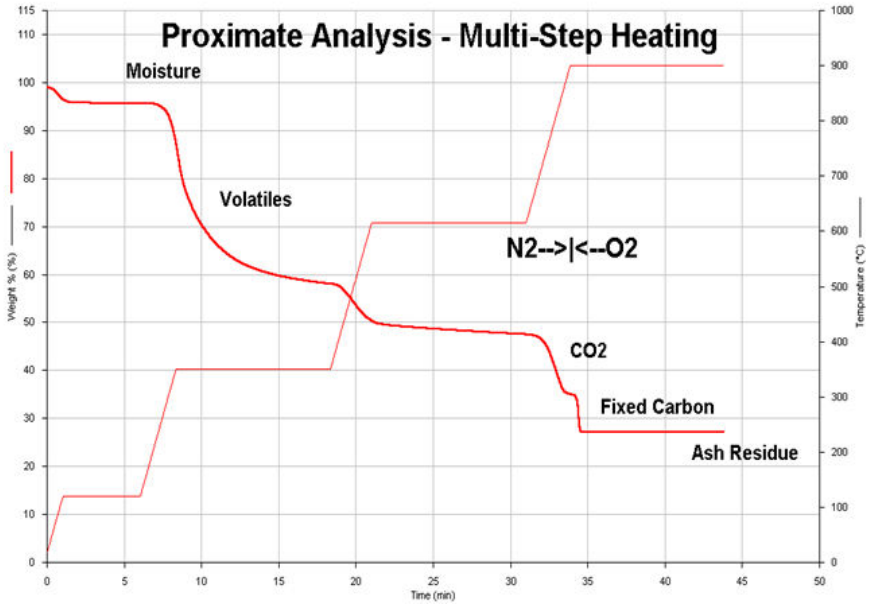
TGA – Thermo Gravimetric Analysis

A *Thermogravimetric Analyzer* (TGA) measures the change in mass of a sample

- Proximate analysis: filler content, carbon black content
- Performance of Stabilizers, Effects of Fillers & Additives
- Decomposition Temperatures
- Oxidation Stability



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Hyphenation
What do we mean by hyphenation?

- Definition
  - A set of instruments connected together to allow more information to be obtained from one run.
    - The name comes from the hyphen used in print to designate the instruments are linked.
- Some well known hyphenated techniques

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Hyphenation
Type of hyphenation with thermal analysis

- Modification of the sample environment
  - UV-DSC
  - UV-DMA
  - %RH-DMA

- Evolved Gas Analysis (EGA)
  - TGA-IR
  - TGA-MS
  - TGA-GCMS
  - TGA-IR-GCMS

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Hyphenation
Who could be interested?

- Polymer and material
  - Polymer (or blend) degradation
  - Material safety and toxicity
  - Polymer identification
  - Nanomaterials
  - Studies of coatings
  - Polymer crystallinity studies
- Pharmaceutical
  - Studies of polymorphs
  - Solvent residues
- Environmental
  - Soil contamination
- Energy
  - Oil and biofuel
  - Solar cell
  - Fuel Cell
- Food
  - Contaminents
  - R&D

Hyphenation – some applications

- TGA-IR
  - Soil analysis
  - Analysis of Layers of a Cable Used in the Automotive Industry
  - Plasticizer Characterization
  - Analysis of Decomposition Products of a Drug
- TGA-MS
  - Residual Solvent Contamination
  - The Analysis of Ethylene Vinyl Acetate
  - High Sensitivity Study of a Solvent of Recrystallization in a Pharmaceutical
- TGA-GCMS
  - Enabling the Analysis of Complex Matrices in Coffee Beans
  - The Analysis of PVC with Different Phthalate Content
  - Qualitative Analysis of Evolved Gases
- TGA-IR-GCMS
  - Unknown aqueous sample

Evolving Gas Analysis (EGA)
Why Studying Evolving Gas?

- TGA tells you when and how much but not what came off

- What came off is important because:
  - It gives you a better understanding of a complex material
  - It explains reaction mechanism
  - It tells you what the reaction by-products are
  - It tells you which solvents are present
  - It allows a greater understanding of the decomposition

Evolving Gas Analysis (EGA)
How Evolving Gas Analysis (EGA) works

- Gas is evolved from a thermal instrument
  - Normally a TGA or STA
  - Gases can be from the evaporation, boiling, or sublimation of solvents
  - Gases can result from reactions, including burning
- A transfer line system moves it to another instruments
  - The line must be inert and heated
  - Temperature must be controlled
  - Somehow the second instrument needs to know the gas is coming across
- A second instrument then measures the components of the gas
  - FTIR allows detection by functional groups
  - MS by mass ion
  - GCMS by chromatography and then MS
- Sometimes a third instrument is added if the previous one is non-destructive.
  - Normally IR followed by MS or GCMS

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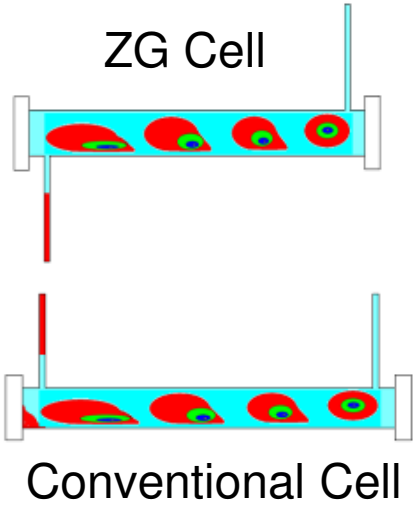
TG-IR
What is needed for TGA-IR?

- Why TGA-IR?
  - Two well known techniques that are complimentary
- Thermogravimetric analyser (TGA or STA)
  - TGA 4000, Pyris One, STA 6000
- FTIR
  - Frontier
    - DTGS detector is usually used
    - An MCT detector could be used for low detection limits
- Transfer line
  - TL-8000
    - Easy to setup
    - Rugged
- Software
  - Timebase
    - Triggered by Pyris (thermal analysis software)
    - Kinetic software

TGA-IR System

TL-8000

- Unique Features
  - Developed for PerkinElmer instruments
  - Easy to setup
    - Plug and Play (TGA and IR)
    - Auto recognition
    - No alignment required
  - Pulling gases with a small vacuum
    - Mass flow controller keep the flow constant
    - Other are trying to push gases thru the small capillary tube…
  - Highest temperature for transfer line and gas cell
    - Prevent condensation
    - Easier to clean
  - The Zero Gravity-effect (ZG) gas cell
    - Reduce cell maintenance
  - Self supported transfer line
    - More reliable results



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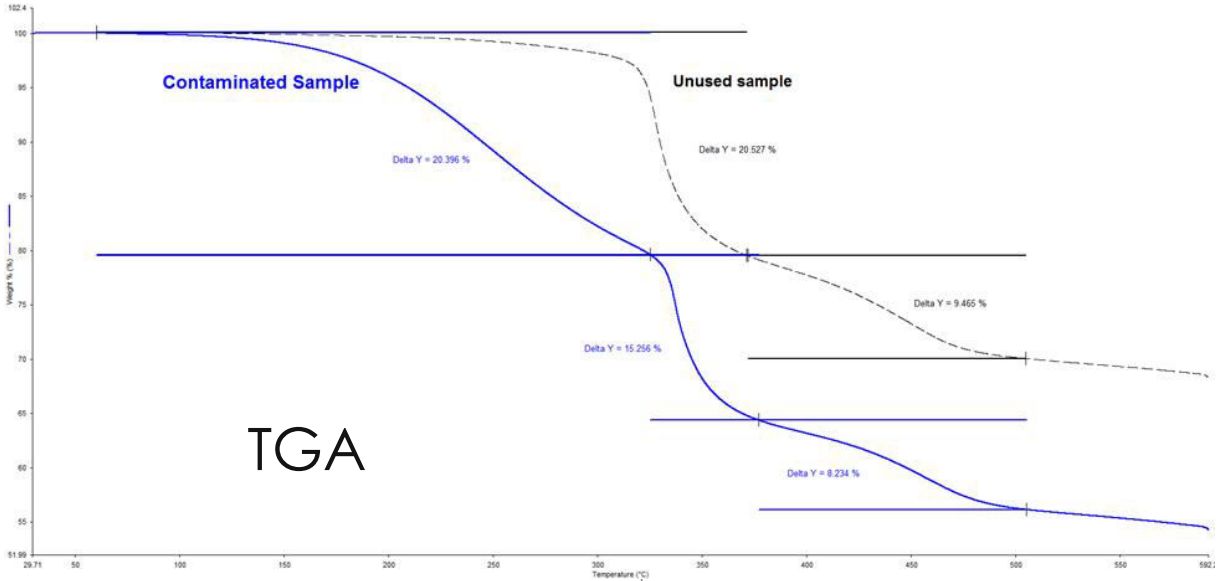
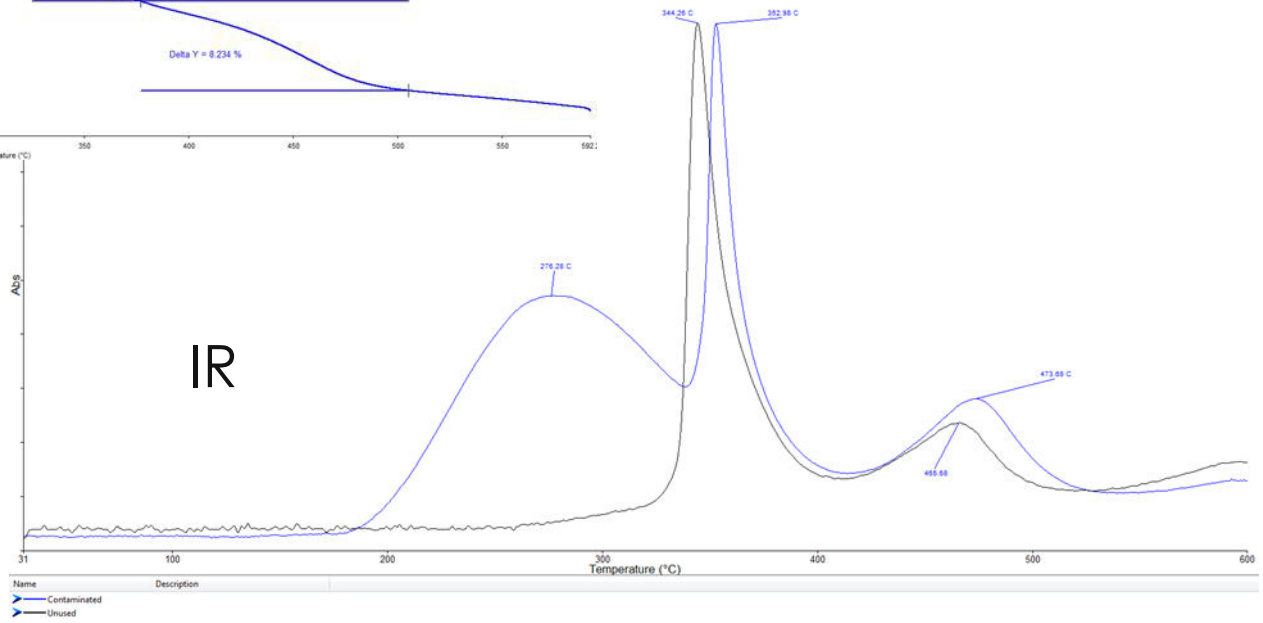
TGA-IR



TGA



IR

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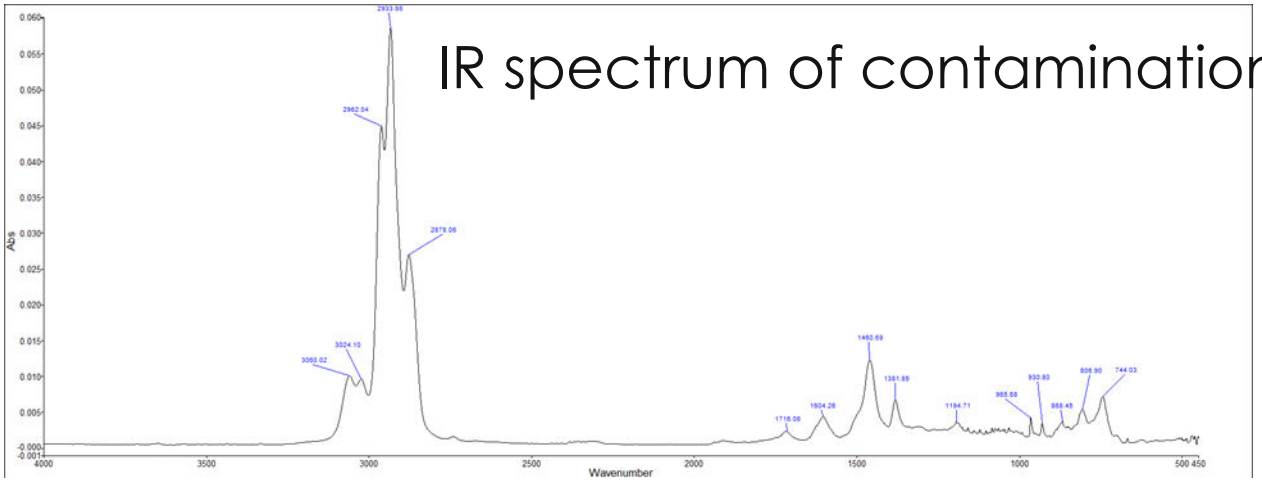
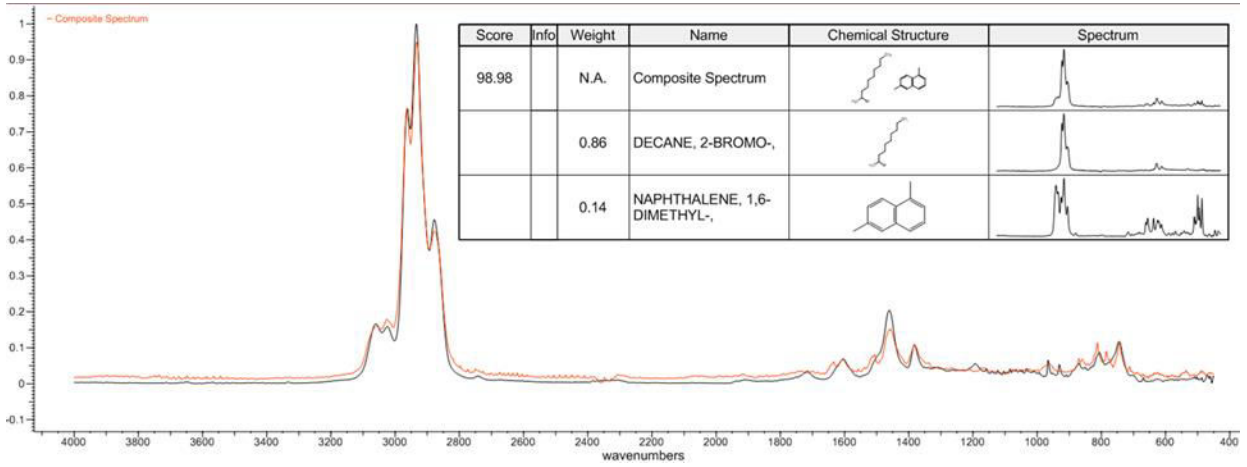
TGA-IR Results



IR spectrum of contamination



Search result

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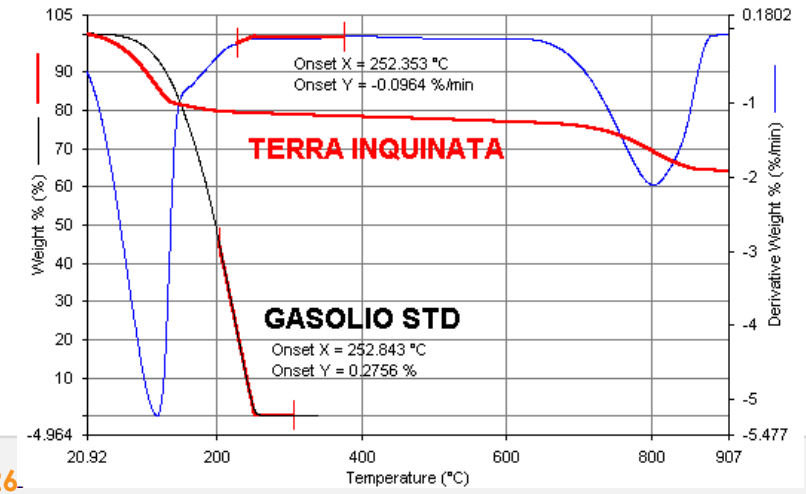
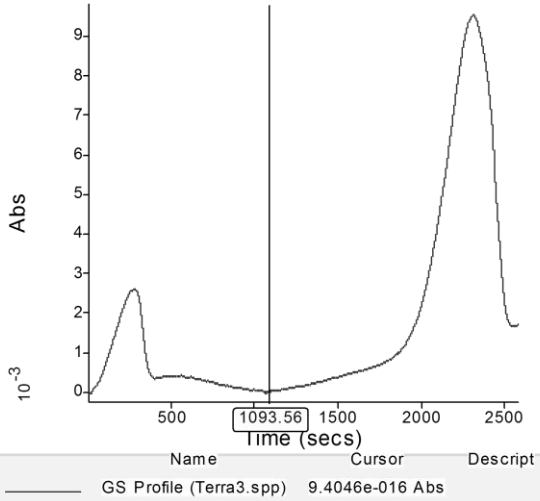
TG-IR Applications
Polymers and Rubbers

- TG-IR
  - Study of polymer (or blend) degradation
    - Weight loss on TGA
    - Analysis of evolved gas on IR
    - Comparison of both technique give extra information
  - Study of material safety and toxicity
    - To know if there is any toxic gases evolving when the material is heated
  - Polymer identification
    - When it is too hard to do it with only FTIR

TG-IR Applications Environmental

- Main application
  - Identification of decomposition product
  - Identification of contaminant
- Example
  - A soil sample might have been contaminated by diesel fuel.
  - Had to find if the sample was polluted and identify the contaminant.
- Experimental
  - Sample: About 30 mg
  - Heating rate: 20°C/min
  - Reference: Diesel fuel
  - Sample preparation: NONE!!





TGA-IR vs TGA-MS

TGA-IR

- Advantages
  - Functional group analysis
  - Vapor phase libraries
  - Allows of structural isomers
  - Real time analysis
  - Qualitative
  - Non-destructive on vapor
  - Lower cost
- Disadvantages
  - Lower sensitivity
  - Difficulties in mixture analysis

TGA-MS

- Advantages
  - Fast analysis times
  - High sensitivity
  - Widely applicable
  - Real time analysis
- Disadvantages
  - More Expensive
  - Limited libraries
  - Could be complex interpretation

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TGA-MS
TGA 8000/Pyris 1 – SQ8

- TGA8000/Pyris 1 TGA
  - SQ8
    - SMART source
  - TL-8500
    - Headspace transfer line
  - Up to 1200 amu
  - Oxygen resistant filament
  - Capillaries of various diameter
  - Soft or chemical ionization
- The only TGA-MS with a unique manufacturer!
- Upgradable to TGA-GCMS!

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TG-MS Applications
Polymers – Analysis of Ethylene Vinyl Acetate

- TG-MS
  - Study of polymer (or blend) degradation
    - Weight loss on TGA
    - Analysis of evolved gas on SQ8 MS
    - Comparison of both technique give extra information
  - Study of material safety and toxicity
    - To know if there is any toxic gases evolving when the material is heated
  - Polymer identification
    - When it is too hard to do it with only FTIR

TGA-MS vs TGA-GCMS

TGA-MS

- Advantages
  - Fast analysis times
  - High sensitivity
  - Widely applicable
  - Real time analysis
  - Quantitative
  - Qualitative
- Disadvantages
  - Could be a real mess!

TGA-GCMS

- Advantages
  - Resolves overlapping events
  - Can use GC libraries
  - Quantitative
  - Qualitative
  - Can use alternative detectors
  - Can use GC techniques to improve separation
- Disadvantages
  - Not Real Time Analysis
  - More Expensive

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TGA-GCMS
TGA8000/P1 TGA – TG-TL-8500-GCMS

- Use PerkinElmer Clarus 600 GCMS
  - Can still do GCMS inside instrument
  - Limited to trapped species so not real time

The best way to detect and indentify small amounts of materials evolved from the TGA.

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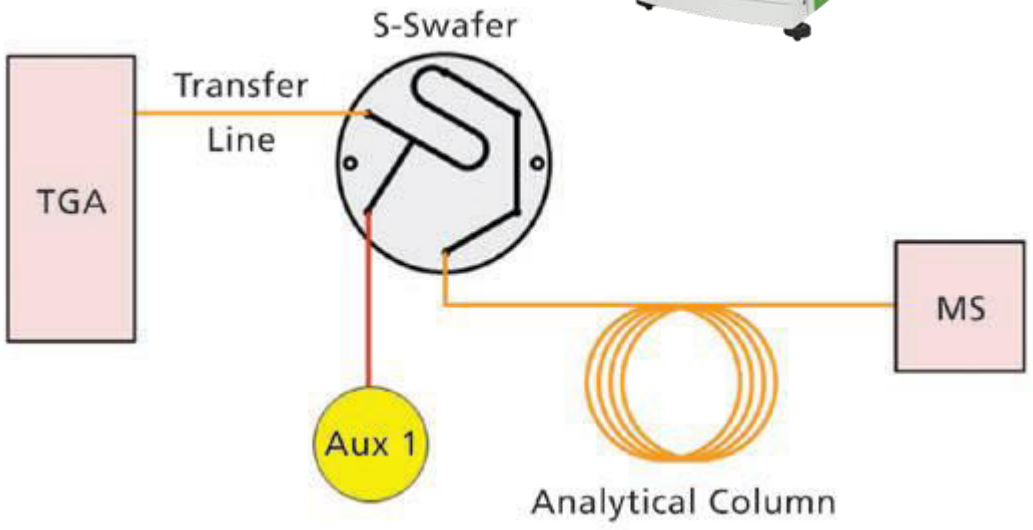
TGA-GCMS
Swafer makes things easier!



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TGA-IR-GCMS

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TGA-IR-GCMS
TGA-GCMS vs TGA-IR-GCMS

TGA-GCMS

- Advantages
  - Resolves overlapping events
  - Can use GC libraries
  - Quantitative
  - Qualitative
  - Can use alternative detectors
  - Can use GC techniques to improve separation
- Disadvantages
  - No real time analysis

TGA-IR-GCMS

- Advantages
  - **Combination of IR and GCMS without the need to split the gas.**
  - Resolves overlapping events
  - Can use GC libraries
  - Quantitative
  - Qualitative
  - Can use alternative detectors
  - Can use GC techniques to improve separation
  - **Can do TGA-IR-MS if real time needed!!**
- Disadvantages
  - More Expensive

TGA-IR-GCMS
TL-9000

- One analysis with three different techniques
  - Results confirmation
- In-line analysis
  - No gas splitting → better signal
- Best detection with TGA-IR-GCMS and best real-time measurement with TGA-IR-MS
  - Get everything you need with one system
- Highest temperature transfer line.
  - Prevent any condensation → don't miss anything
- Self supported transfer line (like TL-8000)
  - Increase results reliability

TGA-IR-GCMS Applications
Analysis of Ethylene Vinyl Acetate

- TGA-IR-GCMS
  - A laboratory often must analyse an unknown mixture to determine the primary components and identify additives or contaminants. This information may be needed, for example, to evaluate a competitor's product or to determine compliance with regulations.
  - TGA, FTIR and GCMS are well known technique for material identification
    - Combining them can give extra information

- Example
  - An analytical lab has received a pigmented aqueous sample for analysis.
  - A complex extraction is usually needed to identify these type of analysis
    - TGA-IR-GCMS removes most of these sample preparation.

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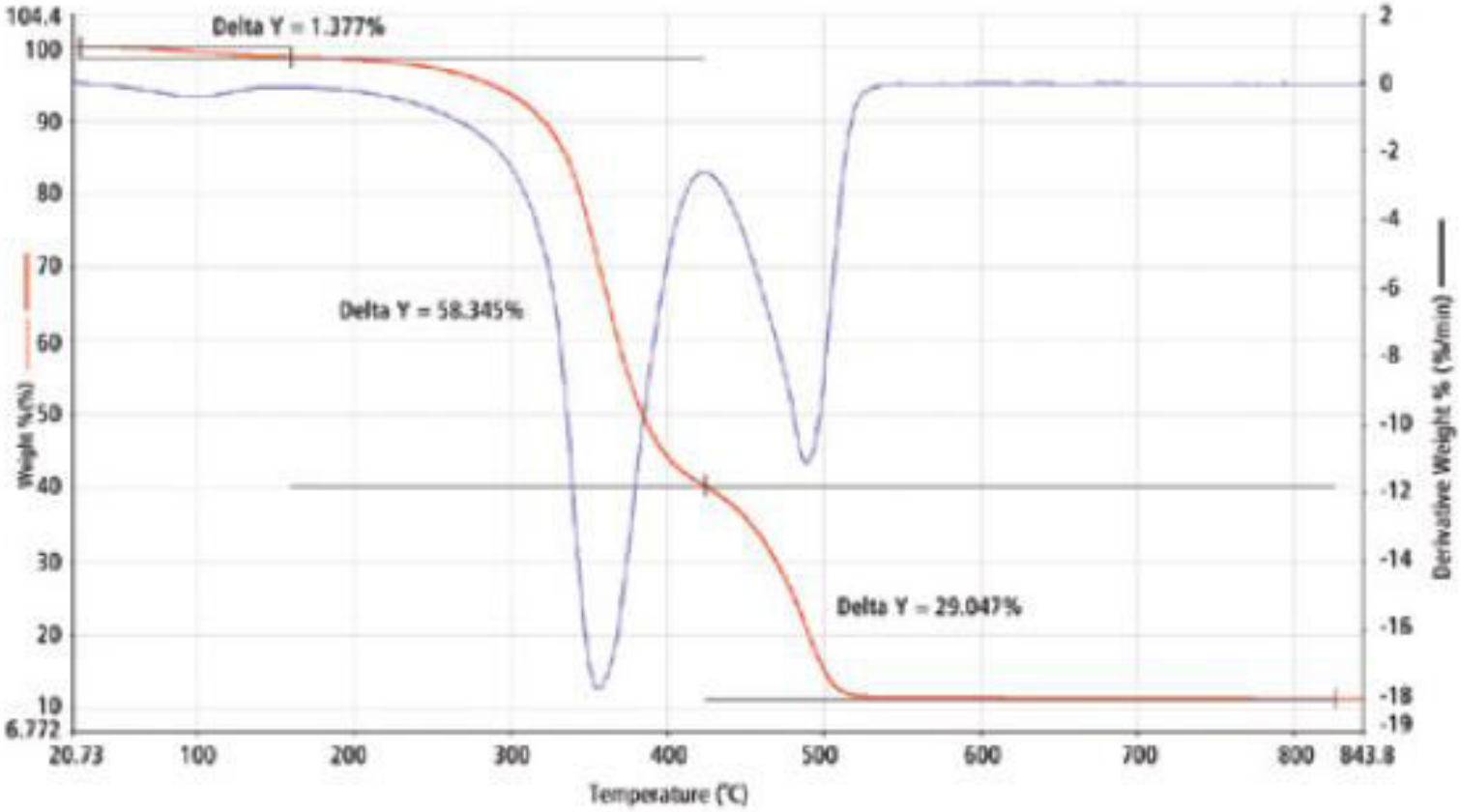
TGA-IR-GCMS Applications
TGA Result



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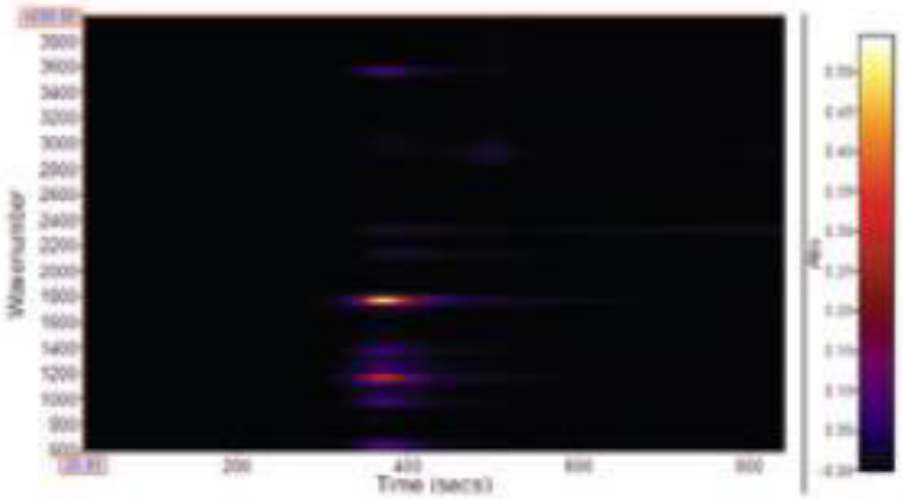
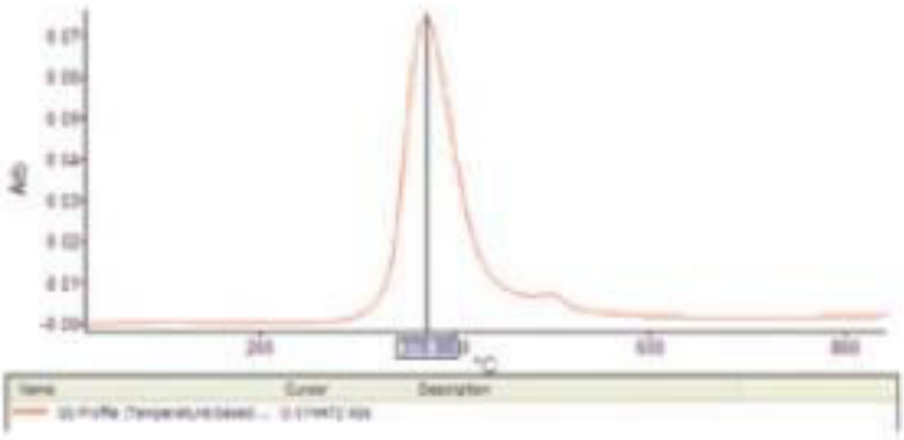
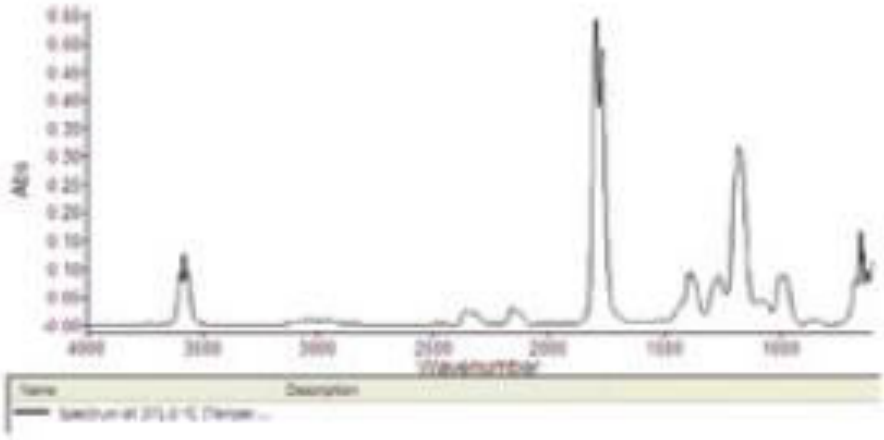
TGA-IR-GCMS Applications
IR Results







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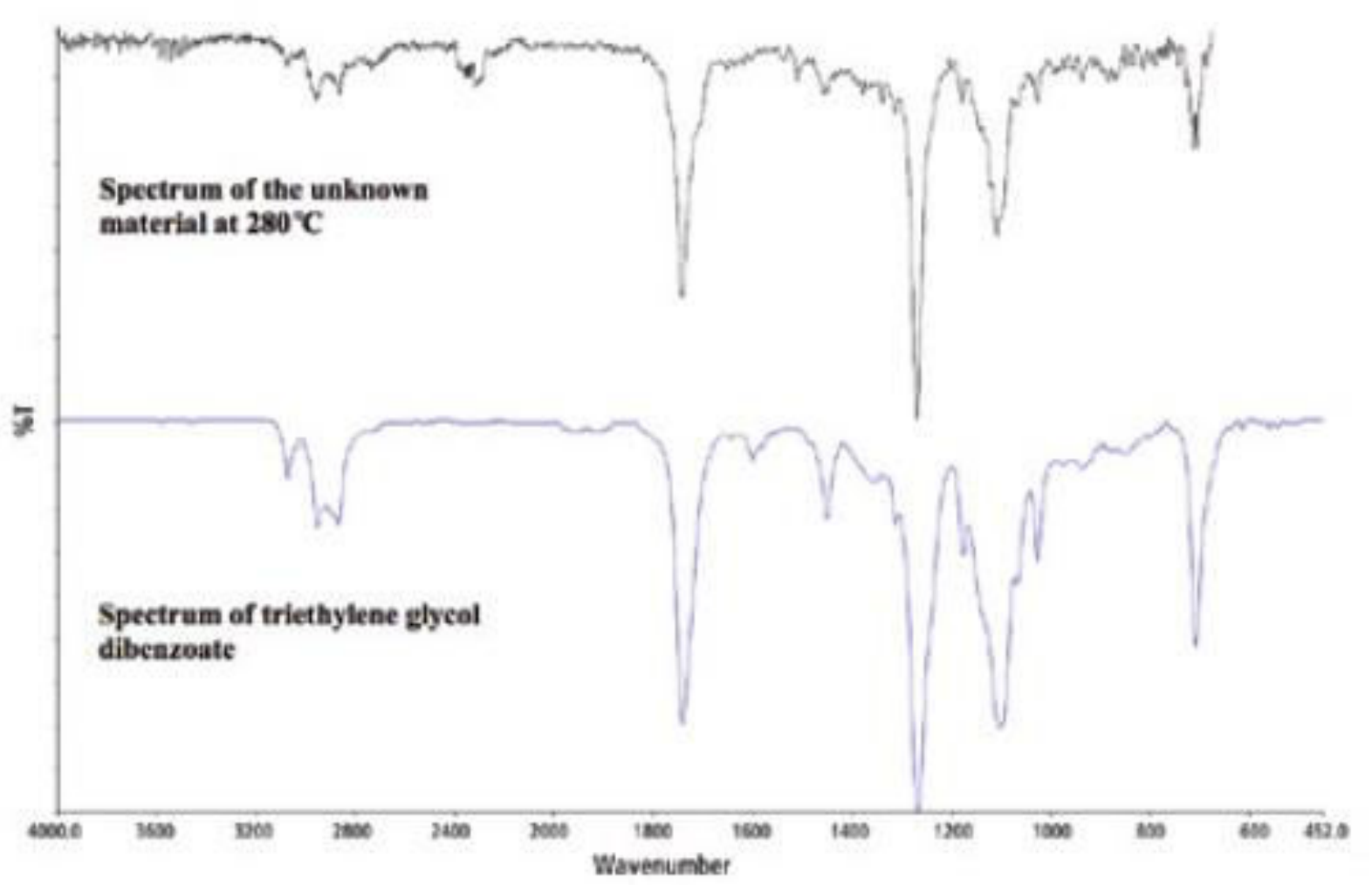
TGA-IR-GCMS Applications
IR Results @ 380C



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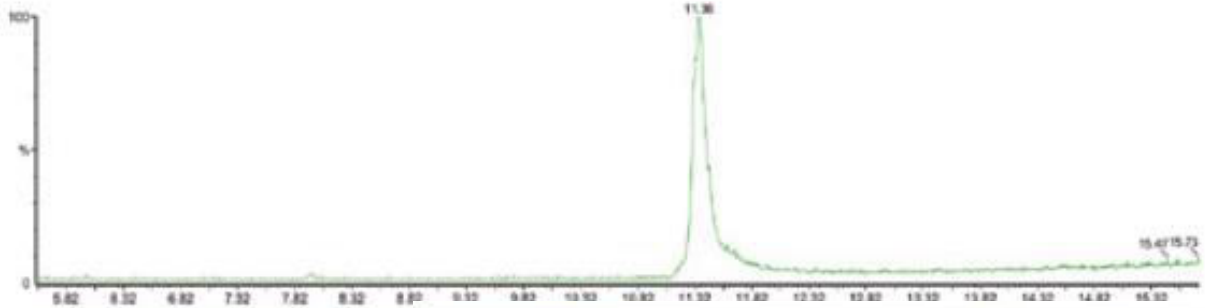
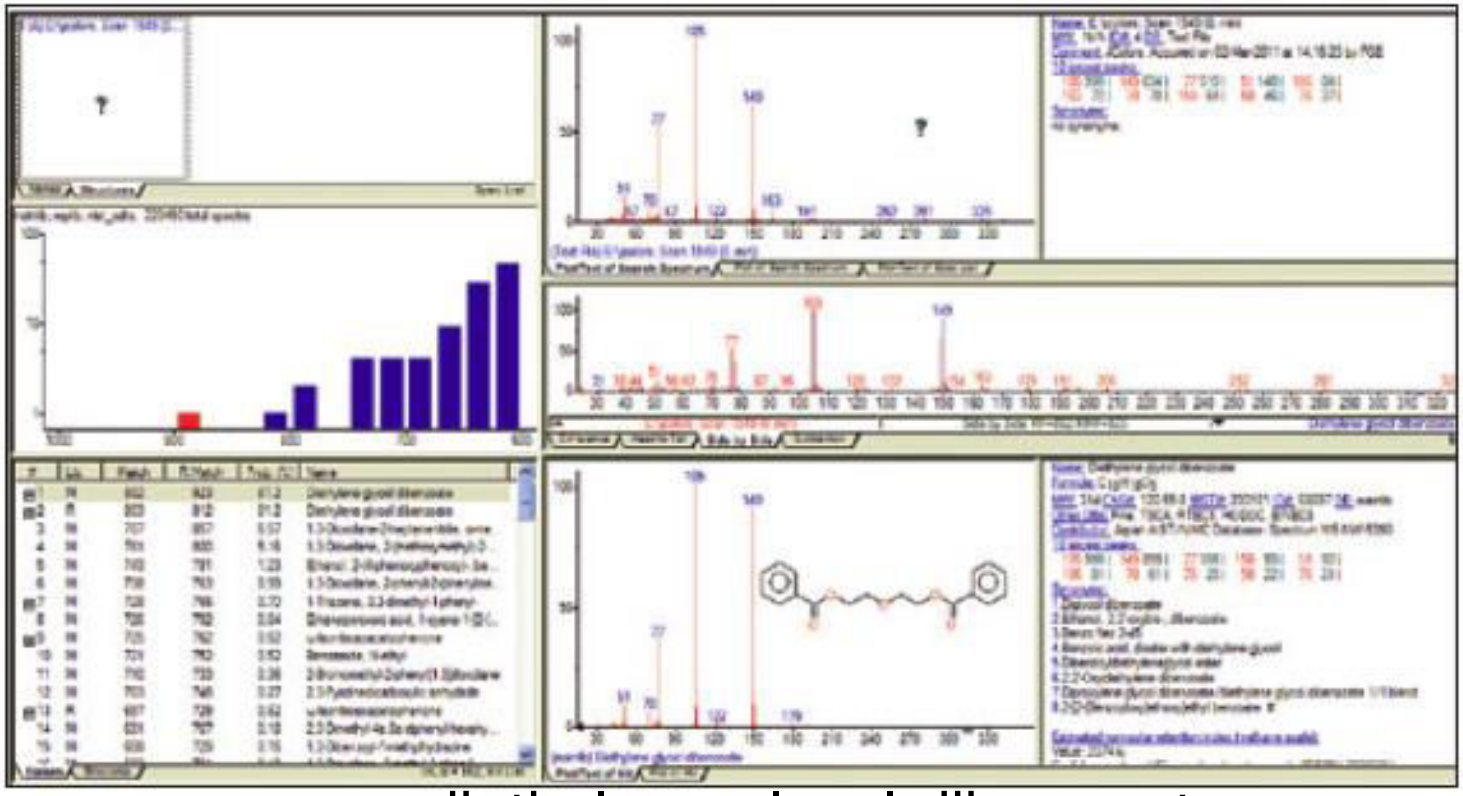
TGA-IR-GCMS Applications
GCMS Results @ 280C





diethylene glycol dibenzoate

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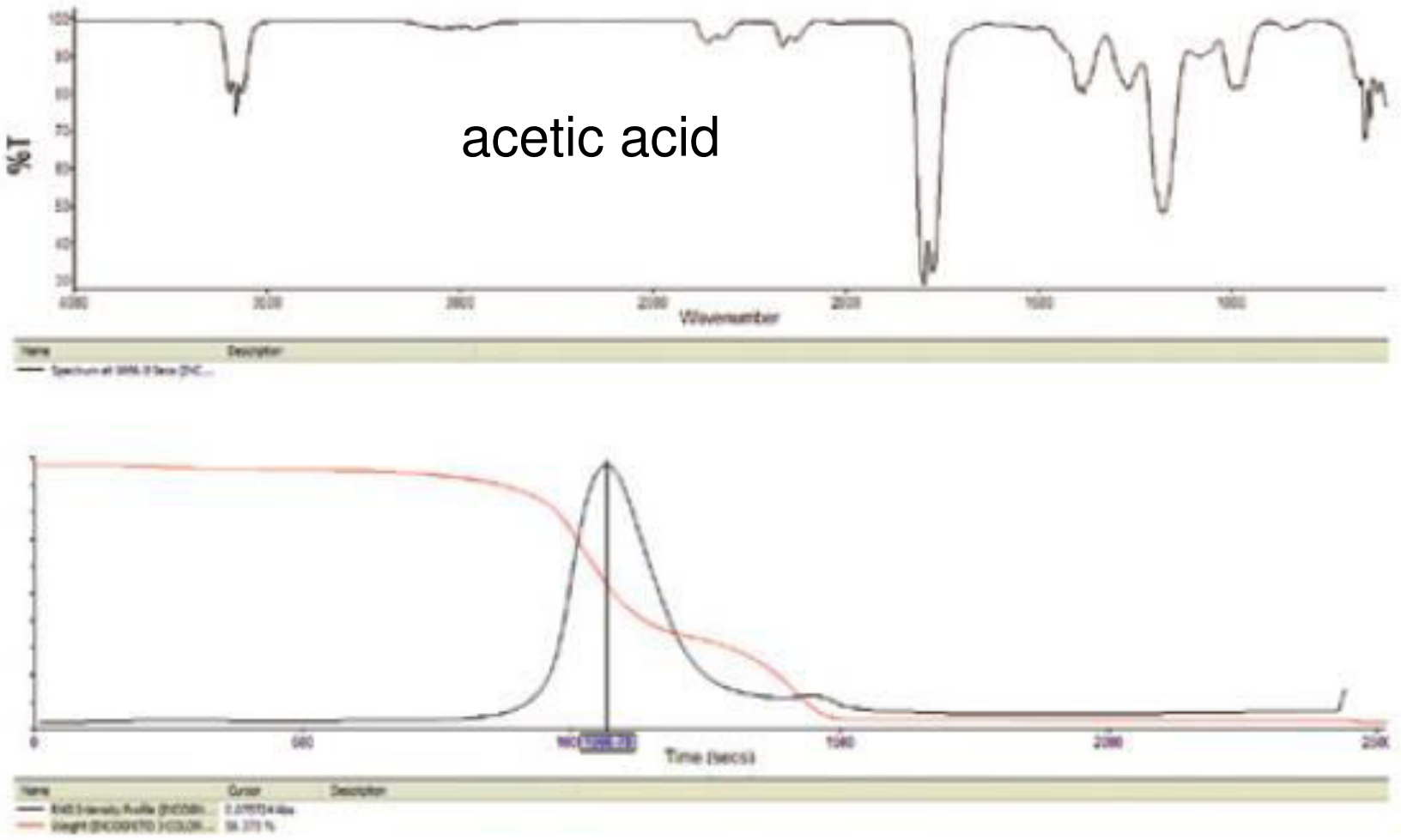
TGA-IR-GCMS Applications
IR Results @ 480C



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Hyphenation
It is not only about the results…!

- PerkinElmer is the only company who can provide a complete solution
  - Thermal Analysis (DSC, TGA, STA, DMA)
  - Molecular spectroscopy (FTIR)
  - Chromatography (GC, GCMS, MS)

- We are the only company who understand every techniques
  - It is easy to get results, not that easy to make sense of them…!

- What about the others?
  - Who takes ownership of the whole system?
  - Who the customers have to call if they have questions?
  - What if something goes wrong with the hyphenated system?

We have THE ONLY Hyphenation Solution – all instruments from ONE Supplier

ULTIMATE PERFORMANCE UNRIVALLED FLEXIBILITY

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