

EVOLVED GAS ANALYSIS (EGA) HYPHENATED TECHNOLOGY GUIDE





THE BEST ANSWERS HAPPEN WHEN GREAT TECHNOLOGIES CONNECT

Introduction

PerkinElmer’s Evolved Gas Analysis (EGA) solutions couple two or more analytical technologies to greatly increase the power of analyses and save precious time by acquiring more information from a single run. PerkinElmer Thermogravimetric Analyzer (TGA) systems coupled with FT-IR (Fourier Transform Infrared), MS (Mass Spectrometry), and/or GC/MS (Gas Chromatography/ Mass Spectrometry) instruments represent the industry’s most complete and advanced line of EGA platforms for materials characterization in polymers, pharmaceuticals, chemicals, petroleum, rubber, food, and more. Increasingly, More applications include identifying harmful chemicals in soil, quantitating components in polymers, determining leachables that may contaminate a product’s packaging, and identifying phthalates in PVC samples.

Hyphenation provides you with the insights to understand when something happened, and also what happened.

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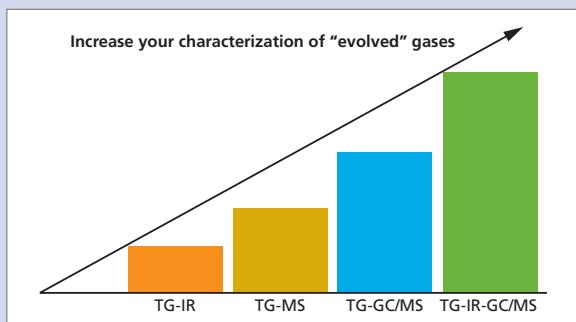
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What Does Hyphenated Technology Mean?

A hyphenated technology describes coupling two or more instruments together to enhance and complement the quality of information. EGA Hyphenation combines a TGA with one or more additional detector technologies, such as FT-IR, MS and/or GC/MS; the most powerful being TG-IR-GC/MS. Hyphenated instruments can combine multiple techniques and acquire additional information from a single run. This is a powerful tool in the analysis and identification of complex mixtures. For example, in the plastics industry, TGA coupled with FT-IR is a common technique used to study the weight loss and chemical components of the evolved gas during material decomposition.

Value of EGA Hyphenation

TGA measures sample decomposition of materials (weight loss event) which provides valuable but limited information. TGA provides quantitative information but does not provide information on what the decomposition products are concurrent to the weight loss. The power of EGA Hyphenation is its ability to carry out in-depth characterization of the "evolved" gases.



Think about plasticizers that are added to polymeric material to increase its flexibility, from those ubiquitous plastic ties to plastic bottles. These plasticizers change many properties of the original polymer and often contain phthalates. Since plasticizers are usually made up of small molecules, they also tend to migrate to the surface and evaporate from the polymer over time or upon heating. Remember that new car smell? More ominous, however, are the apparent health issues associated with plasticizers such as phthalates migrating from some plastic products to humans.

- Provide scientists and manufacturers with a more complete picture of a polymeric material's characterization.
- Leads to better product performance, reduced costs, and improved recyclability.
- Protect consumer health and saves lives. For example, discovery of Bisphenol A (BPA) in plastics and in our foods.

PerkinElmer TGA and simultaneous thermal analysis (STA) systems coupled with FT-IR, MS, and/or GC/MS instruments also represent the industry's most complete and advanced line of hyphenated platforms to help better understand how materials degrade, gases evolve, identifying components/contaminates and how materials respond to a nonstandard test environment like high UV levels or humidity changes. Hyphenation with PerkinElmer's award-winning instrumentation will reveal new information and insights that will provide your laboratory with a competitive advantage that is simply not available to single systems users.

The World Leader in Hyphenated Technology





PerkinElmer has been at the forefront of material characterization technologies for over 80 years. We have been offering total solutions for material research and development, from thermal analysis to a wide range of molecular spectroscopy solutions to the more advanced hyphenated techniques. We can provide the ideal solution no matter what the specifics of your application. We understand the unique and varied needs of the customers and markets we serve. Take it from experience and the only company in the industry capable of manufacturing, supporting, and servicing combined systems that streamline and simplify the entire process from sample handling and analysis to the communication of test results. Hyphenation with PerkinElmer will provide your lab with new pathways for innovation and scientific understanding.

The New E-series Digital Controller

- 7" Touchscreen display
- Easy MFC re-calibration from control panel
- Alert notifications displayed on control panel for immediate diagnostics
- Set pump threshold activation/time prior to shutdown
- Web server enabled



Table 1. Examples of type of hyphenation technologies.

System	Strengths	Applications
 <p>TG-IR</p>	<ul style="list-style-type: none"> • Functional group analyses • Allows structural isomers • Real time analysis • Qualitative analysis • Non-destructive on vapor • TG and IR can easily as a standalone instrument (if desired) 	<ul style="list-style-type: none"> • Industrial – QA/QC, Thermal Stability, Bio-polymers gases degradation • Food – adulteration • Environment – contaminated soil • Pharmaceutical – residual solvent • Unknown identification • Decomposition studies • Polymer QA/QC
 <p>TG-MS</p>	<ul style="list-style-type: none"> • Fast analysis times • High sensitivity • Widely applicable • Real time analysis • Quantitative analysis • Can use MS library 	<ul style="list-style-type: none"> • Industrial – nanomaterials, polymer additives, process optimization • Food – adulteration, food quality and packaging • Environment – contaminated soil, trace amount • Pharmaceutical – residual solvent, solvates: crystal shape evaluation (solvent-molecule binding energy)
 <p>TG-GC/MS</p>	<ul style="list-style-type: none"> • High sensitivity • Resolved overlapping events • Quantitative analysis • Qualitative analysis • Can use alternative detectors • Improve separation by GC approaches • Can use MS library • FAST GC capable with Clarus 690 	<ul style="list-style-type: none"> • Industrial – QA/QC, nanomaterials, polymer additives, process optimization • Food – adulteration, food quality and packaging • Environment –contaminated soil, trace amount • Pharmaceutical – residual solvent, solvates: crystal shape evaluation, excipients compatibility, shelf life, and thermal degradation ensure proper spacing
 <p>TG-IR-GC/MS</p>	<ul style="list-style-type: none"> • Functional group analysis • Vapor phase libraries • Allows structural isomers • Real time analysis • Qualitative analysis • Quantitative analysis • Non-destructive on vapor • Fast analysis times • High sensitivity • Widely applicable • Resolved overlapping events • Can use alternative detectors • Improve separation by GC approaches • Can use GC library • TGA, IR and GCMS can easily be used independently • FAST GC capable with Clarus 690 	<ul style="list-style-type: none"> • R&D Material Characterization, solvent – molecule binding energy, state transitions • Industrial – competitive analysis, nanomaterials, polymer additives, process optimization • Food – fats and oils, adulteration, carbohydrate analysis, state transitions, additives, food quality and packaging • Environment – contaminated soil, trace amount • Pharmaceutical – residual solvent • Unknown identification • Decomposition studies • Pharma - Drugs: Solid state characterization (polymorphism/pseudopolymorphism), solvates crystal shape evaluation, excipients compatibility, shelf life, and thermal degradation • Battery research on lithium polymer (LiPo) electrolyte (SPE) and polymer separator degradation • Graphene and CNT based batteries, proof of successful hybridization • Kinetic Analysis, i.e. pyrolysis, catalysis etc. • Fire Retardation (performance evaluation and identification) • Chemical Identification • Petroleum, Lubricants, and coal Industry Research

Experience Better and Reproducible Results with a State-of-the-art Interface.

PerkinElmer offers interfaces that can be connected between various FT-IR*, GC/MS**, TGA*** and STA***, offering you a broad range of options in terms of price and performance to fit your needs.

Key Advantages:

Highest temperature up to 350 °C to avoid condensation and deposition of evolved gases.

Replaceable internally treated liner for an easy maintenance.

Zero-Gravity Cell (ZG-Cell):

As shown in Figure 1, design allows heavy molecular weight components elimination, providing the cell with little maintenance and more sensitive and accurate data. ZG-Cell is provided with an automatic accessory identification, low volume and efficient sample area purging.

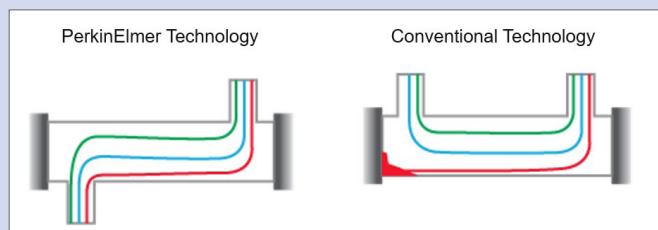


Figure 1. Zero Gravity Cell Technology.

Temperature ramp in transfer line

- Reduces the risk of a secondary degradation
- Volatile sample integrity is preserved

Gas Stream Filtration

Gaseous samples are filtered before going into the FT-IR and GC/MS instruments.

Mass Flow Controller:

The interface controls the flow instead of the pressure. In analytical terms, this means quantitative results for the detection as well as reproducible analysis without stagnation of corrosive gas in the TGA furnace.

Flow Pressure

Balanced Flow Interface ensures:

- A constant pressure inside the TGA furnace
- Precise flow and stable setup temperature
- No stagnation of corrosive gas in the furnace

Triggers

Maximize your control with ultimate trigger control

- Define multiple unique trigger points
- Set multiple trigger points sequentially (i.e. for FAST GC)
- Maintain an isothermal run with multiple trigger points (i.e. for collecting a degradation profile for specific compounds)
- Use multiple trigger points to increase the sample amount and/or sample composition on the GC column by keeping the column isothermal (i.e. for simulated pyrolysis)
- Multiple triggering possibilities: i.e.
 - TGA Trigger GC Mode: Time, Temperature or Weight loss
 - TGA Trigger Online mode: OnLine experiment (TGA-MS)
 - TGA Trigger TAB mode: Time table of a saved method, or manual run as you do TGA run (will record) FT-IR trigger

Active Hyphenation versus Passive Hyphenation

Many systems use passive hyphenation that can result in (a) sample destruction, resulting in the loss valuable data from the FT-IR and GC/MS instruments. (b) Damage to the TGA furnace itself (c) even risk of injury to personal using the instrument

PerkinElmer uses Active Hyphenation that draws gas partially from the TGA furnace leaving the lowest dead volume, for better performance and data with less maintenance.

Dead Volume

Active hyphenation system with the lowest dead volume (<2.5 ml) compared to the other TGAs in the market.

Dead Volume is the unmoving, stagnant or un-swept gas trapped in the furnace space. Over time the stagnant gas deposits over the surfaces and can deleteriously affect the TGA balance mechanism creating problems to the TGA normal functionalities.

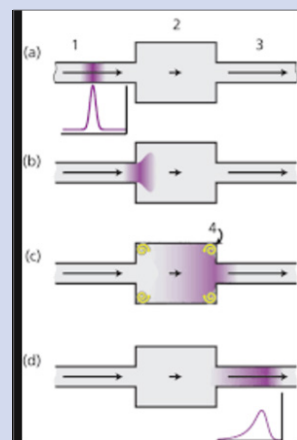


Figure 2. Dead Volume and Flow Pressure in EGA hyphenation.

* Spectrum Two, Spectrum 3, Frontier, Spectrum 65/100/400/One series

** Clarus GC and SQ8 MS

*** TGA 8000, TGA 4000, STA 6000, STA 8000, Pyris 1 TGA



The combination of a TGA with TG-IR is the most common type of EGA system in use today. The gases evolved during the decomposition of samples are transferred to an IR gas cell, via specialized heated transfer lines, where the components can be identified. Because of its ability to detect functional groups, gas-phase IR analysis allows greater understanding of the processes seen in the TGA.

Unlike other systems that simply move the gas to the TGA, the state-of-the-art PerkinElmer TG-IR system is designed to ensure every component evolved in the TGA is transported to the IR.

Advantages of PerkinElmer TG-IR System Include:

- Insulated heated transfer line with replaceable SilcoSteel® liner
- Heated zero-gravity-effect 'ZGCell' gas cell for the PerkinElmer FT-IR instrument incorporating automatic accessory identification, low volume, and efficient sample area purging
- Control unit incorporating a mass flow controller, particle filters, flow smoothing system, independent transfer line and gas cell temperature controllers, and vacuum pump with exhaust line
- Automatic triggering of IR data collection from the Pyris™ Software that controls the TGA
- Spectrum Timebase™ Software for time-resolved experiments
- Constant gas flow giving optimum separation between time-resolved peaks
- Reduced mixing of IR signals
- No accumulations of heavy components in the IR cell due to ZGCell design
- Automatically import TGA data in Timebase Software included in Spectrum 10 software enabling easy to use compare function

The TG-IR transfer line can be used to connect any of the PerkinElmer TGA/STA instruments to any of our FT-IR's. This offers a range of options for performance to fit your needs.

The TG-IR system is ideally suited for applications identifying materials evolved on heating, like residual solvents in pharmaceuticals, component identification in the analysis of plastics or rubbers, or the study of the combustion products from burning a sample. An example of the data one can get is shown below from a sample of switchgrass, a material being studied in North America as a possible source of biofuels. As shown in Figure 1, there is a small weight loss at low temperatures and then a much larger one at approximately 250 °C, which corresponds to the burning of the organic matter. This data is imported into the Timebase Software where it can be

compared to the total absorbance curve and the area of interest selected for analysis. Here we select 30 minutes in the midst of the burn and look at the spectra of the evolved gases.

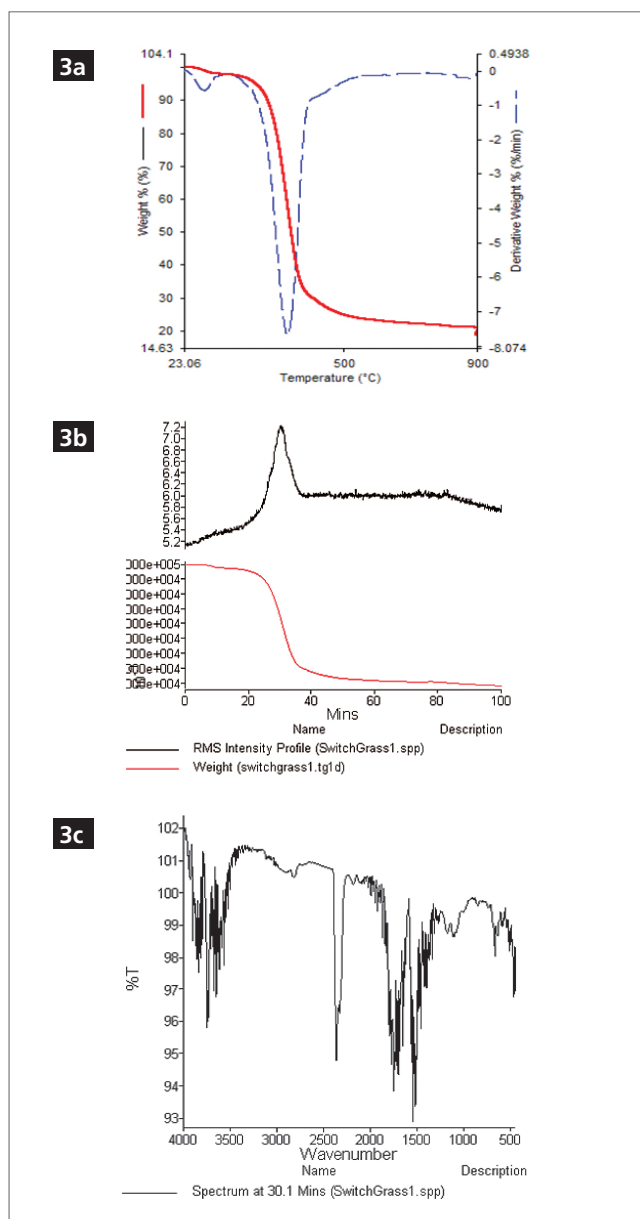


Figure 3. The data from the TGA run (top 3a) is transferred automatically to the Timebase Software and compared to the Gram Schmidt plot (middle 3b). From this data, we can examine regions of interest as shown in the lower center image (3c).



The PerkinElmer TG-MS transfer line allows you to couple our full range of Thermogravimetric Analyzers (TGA) and Simultaneous Thermal Analyzers (STA) to a PerkinElmer Clarus® SQ 8 GC/MS.

The TG-MS system uses:

- Our range of TGA and STA Systems, to optimize sensitivity to weight loss
- The TG-MS is equipped with a transfer line with adjustable heating capabilities up to 350 °C, mass flow control, and pumps. The TG-MS allows connection to other brands of MS
- The Clarus SQ 8 Mass Spectrometer, for accurate identification

By using the PerkinElmer Clarus SQ 8 Mass Spectrometer, the same MS used in PerkinElmer's state of the art GC/MS systems, enjoy the advantages of:

- The detection of mass ions up to 1200 daltons
- Soft ionization (adjustable EI) to limit fragmentation of the mass ion
- The ability to add chemical ionization (CI) to decrease fragmentation
- Automatic triggering of the MS run at the start of the TGA run

One of the advantages of TG-MS is it is real time and very sensitive. This has several applications in detecting residual solvents in pharmaceuticals and in measuring additives in polymers. In Figure 4, we see an example of a TGA curve, overlaid with the mass ion peaks for three common solvents.

In Figure 5, we see the decomposition of rubber in the TGA while the evolved gases are tracked as a function of time, enabling further data and analysis.

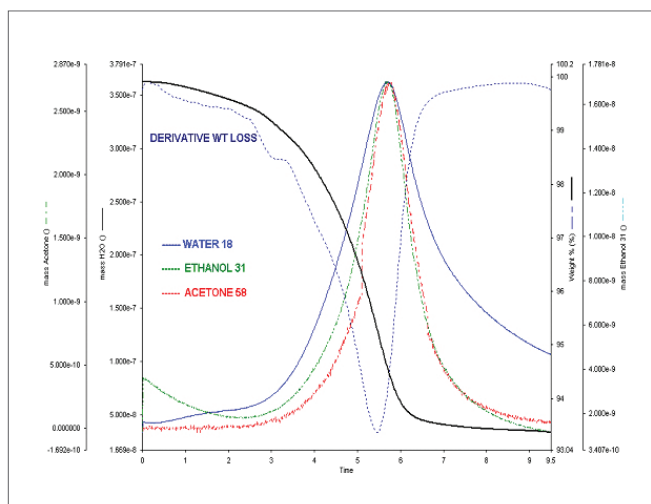


Figure 4. TG-MS enables the quantification of the weight loss from a material and identify the components being evolved.

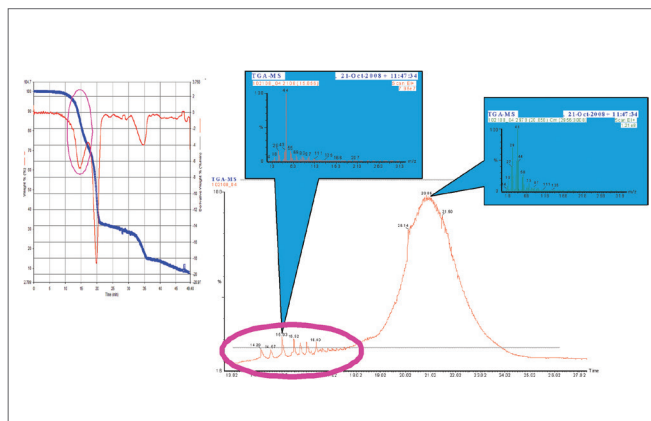


Figure 5. When burning a complex material like rubber, the TGA data can be limited. Facilitating the analysis of the evolved gases through MS enables further analysis. The TG-MS showed what materials are being lost with temperature in rubber.



Benefits of FAST GC:

- Faster results with time savings up to 80%
- Analyze multiple events in a single run
- Double event separation
- Automatic triggering by time or weight loss

Although TG-MS allows real-time monitoring, it can be confusing due to overlapping events and higher mass ions obscuring those of lower mass. By adding gas chromatography (GC) to the system, these events can be cleanly separated and very low levels of impurities detected.

Heating a sample using the TGA causes a sample to release volatile materials or generate combustion components as it burns. These gases are then transferred to the GC, where the components can be collected on a trapping media, in a gas sampling loop, or deposited on the head of a column. The sample can then be run by GC to separate the material, and the peaks identified by the MS. Because of its ability to detect very low levels of material in complex mixtures, the TG-GC/MS is a powerful tool for quality control, safety, and product development. The TGA is acting like Thermal Desorption, Headspace or Pyrolysis but with controlled active purge gas. Unlike a pyrolysis unit, the TGA tells you how much has come off, how quickly and at what temperature.

- Any of the TGA 4000, STA 6000, STA 8000, Pyris 1 TGA or TGA 8000 can be connected depending on degree of precision and temperature range needed.
- Switch from GC separation (TG-GC/MS mode) to Single Ion Monitoring (TG-MS mode) quickly and easily via software alone.
- The TG-GC/MS transfer line runs at 350 °C and uses pumps and mass flow controllers to deliver a precise flow of gas to the GC/MS. Two sample collection loops are included.
- Clarus SQ 8 GC/MS provides maximum detection of low levels of contaminants. detect components traces at the level of part per billion.

Coupling the PerkinElmer TGA 8000 to the Clarus SQ 8 GC/MS allows several options to collect the evolved gases, including the ability to set various trigger points. Of these, the use of the chilled oven to collect the volatiles on the top of the GC column has been found to be most convenient.

A small quantity of dried and ground switchgrass was placed on the TGA pan and weighed using the Pyris software. A rapid TGA analysis based on heating the sample from 30 °C to 1000 °C at 100 °C/min in a nitrogen atmosphere was performed to determine which regions of the weight loss curve were to be further studied using the TG-GC/MS technique.

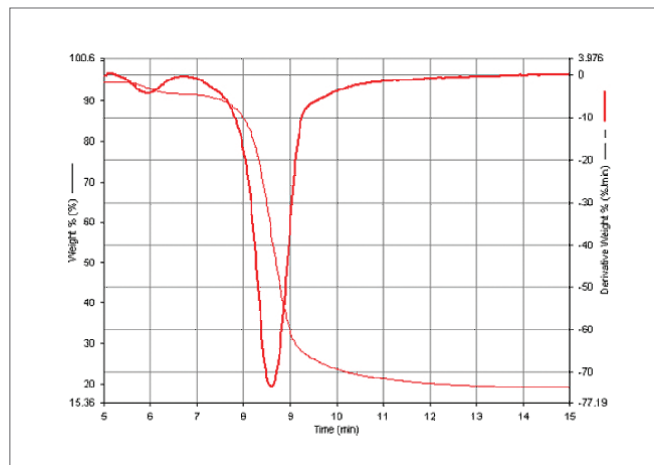


Figure 6. The TGA run of a sample of switchgrass shows most weight loss occurs in one temperature range.

Collecting the material on a GC column and eluting it gives the following chromatogram. Using the data from the MS to identify the peaks, the progression of acids can be detected easily in the offgas. These components cannot be isolated by TG-IR or TG-MS due to their low levels and the complexity of the mixture.

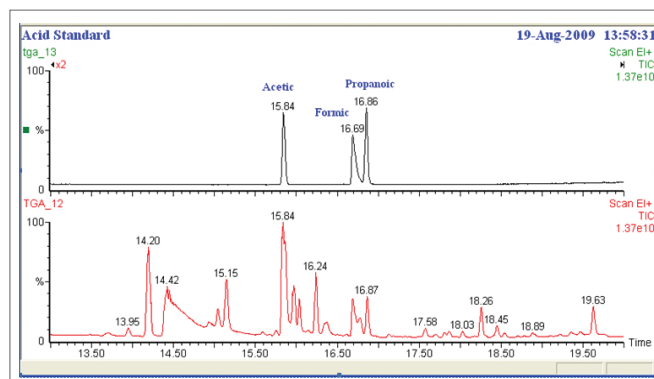


Figure 7. GC/MS on the gases evolved between 8 and 9 minutes and collected on the head of a GC column gave the chromatography seen on the bottom of the graph. MS analysis suggest that 15.8 is the acetic acid, which is confirmed above by running a standard of acetic, formic, and propanoic acids.



Hyphenating TG-IR-GC/MS is a powerful approach for analysis of an unknown mixture to determine its 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.

The PerkinElmer TG-IR-GC/MS transfer line is used to allow TG-IR-GC/MS analysis on a sample by moving the off gases to the FT-IR and GC/MS after their evolution in the TGA. It acts as the interface between a TGA or STA, an FT-IR like the Frontier or Spectrum Two FT-IR, and a Mass Spectrometer or GC/MS, such as the Clarus SQ 8.

During the TGA thermal separation of a pigmented aqueous sample, the gases released by the sample were sent to the FT-IR for spectral analysis. The TG-IR data consists of a sequence of spectra, acquired at intervals of around 8 seconds. The standard presentation of the data is the adsorption versus wave number, and this spectral profile of the gases released by the sample is generated for each at roughly a two-degree interval. The TG-IR Spectrum Time Base Software provides a 3D graphical representation, consisting of stacked IR spectra, a feature that provides a snapshot of the entire TG-IR separation.

The TG-IR-GC/MS interface was used to perform a subsequent analysis to confirm the identity of the unknown substance in the aqueous sample. At the time of maximum concentration absorbance of the substance being analyzed, the gas in the IR gas cell was sent to a GC/MS.

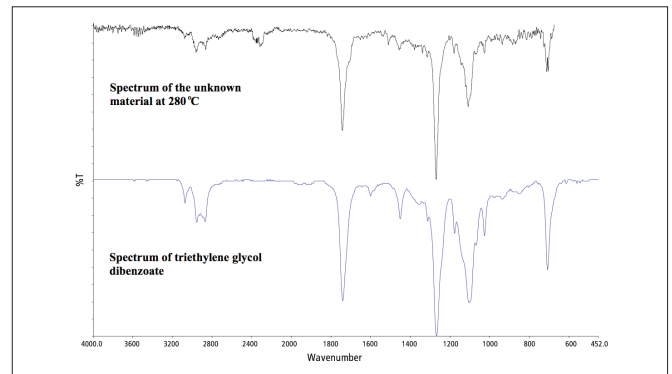


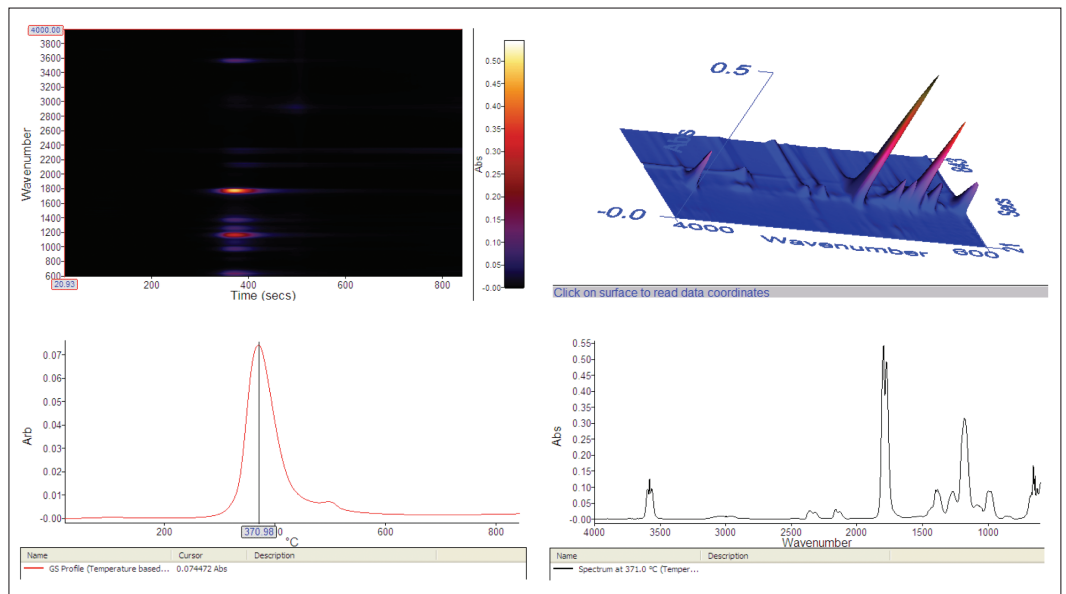
Figure 9. Best match spectra using PerkinElmer Spectrum Search software.

Our search software provides you with instant candidate matching information at the touch of a button.

Advantages

- TG-IR-GC/MS allows one to gain the advantages of two EGA techniques and compensate for their disadvantages.
- Unlike TGA-GC/MS, the TG-IR-GC/MS retains the relationship between temperature a gas is released at and its components by FT-IR.
- The sequentially measuring of the offgas components by GC/MS allow detection of trace levels too low to be seen in the FT-IR.

Figure 8. Spectrum Time-Based software outputs, which aid in interpreting the decomposition process. With experience, an operator will look at the stacked spectra (upper right plot) and see an “unexpected mountain range” that represents the transient presence of a particular species of off-gassing product of potential interest.



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Protect Your Instruments with PerkinElmer OneSource[®] Laboratory Services

Now that you've made an investment in new instrumentation for your lab, it's time to protect it. OneSource Laboratory Services can help with routine maintenance and repair service from our team of certified engineers along with expert solutions and carefully engineered programs designed to achieve your best targeted business outcomes.

With over 15 years' of experience and a proven track record of success, OneSource has a deep knowledge and understanding of laboratory and R&D needs and offers a one-of-a-kind systems approach to success. Through insight and expertise OneSource consultants can pinpoint specific issues that impact efficiencies across all facets of operations, and provide innovative, holistic solutions to address them.

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- Instrument Service and Repair
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- Laboratory IT Services
- Asset Data Visualization Tools
- Laboratory Efficiency Consulting
- Scientific Staffing Solutions



Markem-Imaje Corp. is a worldwide leader of product identification equipment, software, supplies and services for marking and coding. Markem-Imaje has a very active and busy material characterization laboratory, managed and run to conduct new product research and solve day-to-day product problems.

“Whether I am searching for a new technique or in need of enhancing a current instrument to serve my industry better, PerkinElmer’s willingness to engage our needs enforces our belief that they are the business partner of choice in material characterization. I would like to thank your entire team for being that business partner that is helping me succeed in helping my company succeed.”

– Customer Testimonial from Samuel Apkarian, Markem-Imaje Corp.

THE BEST ANSWERS HAPPEN WHEN GREAT TECHNOLOGIES CONNECT

For more information, visit <http://www.perkinelmer.com>

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