

NETZSCH



Leading Thermal Analysis ■

Coupling to Fourier Transform Infrared Spectrometers FT-IR

Concepts, Instruments and Applications from (10°C) RT to 2000°C

Thermal Analysis Plus FT-IR

More Than Just the Sum of Its Parts



FT-IR
Coupling

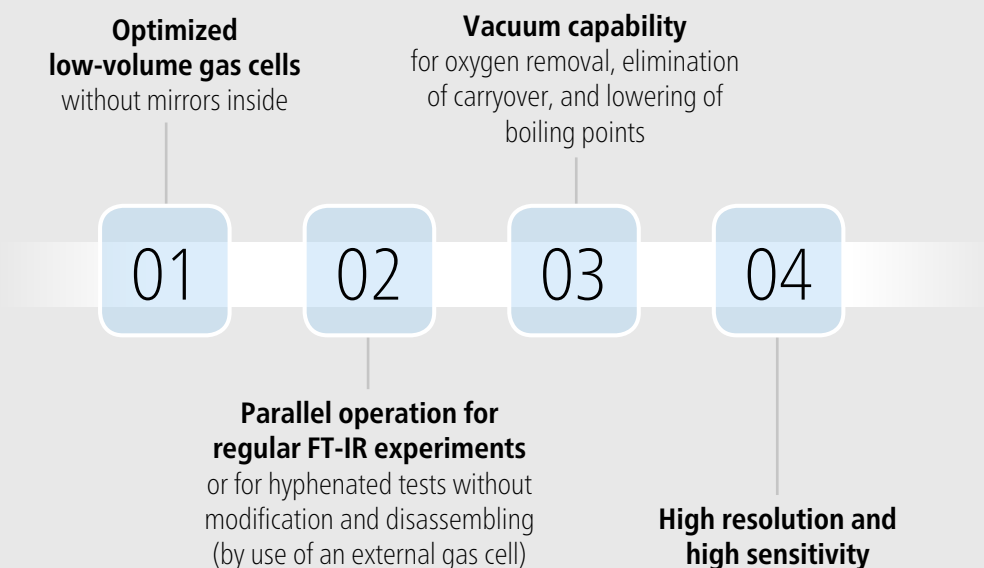
Perfect Combination Bridges the Analytical Gap and Facilitates Fingerprint Analysis

Thermal Analysis provides ideal tools for the characterization of all kinds of organic and inorganic solids and liquids. Thermodynamic transitions, thermal stability, decomposition and chemical reactions can be detected and quantified with high accuracy over a broad temperature range.

In some cases, however, information about the type of gases evolved is needed in order to gain a detailed understanding of the chemistry behind the processes. The combination of thermal analysis with the powerful infrared spectroscopy for gas analysis bridges this analytical gap. It allows for deeper insight into the material's behavior and may provide a fingerprint of the analyzed material.

The *Proteus*[®] software for thermal analysis and the OPUS software for the FT-IR are **integrated with one another** to support the Thermal Analysis-to-FT-IR coupling. The relationship to temperature and time of all information produced by the running experiment is meticulously maintained.

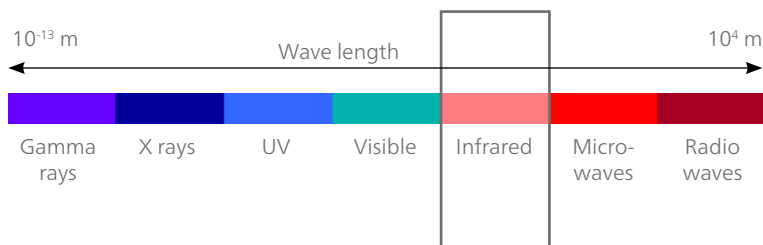
Harmonized Instrument Combinations with Fully Integrated Software from the Specialists in Thermal Analysis and IR Spectroscopy



Great Variety of Configurations to Meet all Needs

Coupling type	Transfer line – external gas cell	Transfer line – internal gas cell	FT-IR-MS	FT-IR-GC-MS	PERSEUS® Coupling
TG 209 F1 <i>Libra</i> ®	✓	✓	✓	✓	✓
TG 209 F3 <i>Tarsus</i> ®	✓	✓			
STA 449 F1/F3/F5 <i>Jupiter</i> ®	✓	✓	✓	✓	✓ (F1, F3)
STA 2500 <i>Regulus</i> ®	✓	✓	✓	✓	
DSC 204 F1 <i>Phoenix</i> ®	✓	✓	✓	✓	
DSC 404 F1/F3 <i>Pegasus</i> ®	✓	✓	✓	✓	
TMA 402 F1/F3 <i>Hyperion</i> ®	✓	✓	✓	✓	
DIL 402 <i>Expedis Select/Supreme</i>	✓	✓	✓	✓	

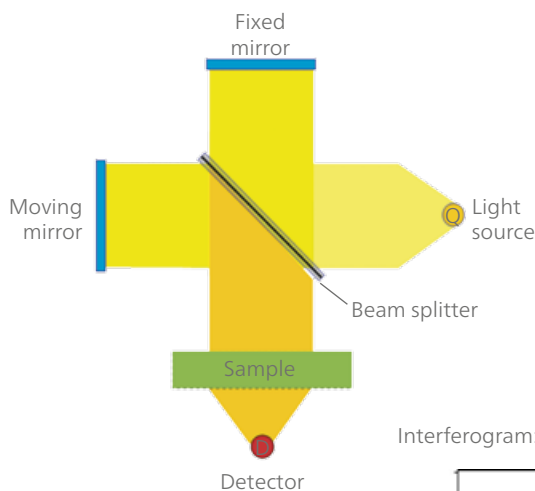
Optimized TGA/STA/DSC – FT-IR Coupling



Infrared spectroscopy is a classical technique which depends on the interaction of infrared radiation with the vibrating dipole moments of molecules. It provides a characteristic spectrum for each substance, with the exception of homonuclear diatomic molecules or noble gases. Examples of homonuclear diatomic molecules are N_2 and O_2 .

Functional Principle of an FT-IR Spectrometer

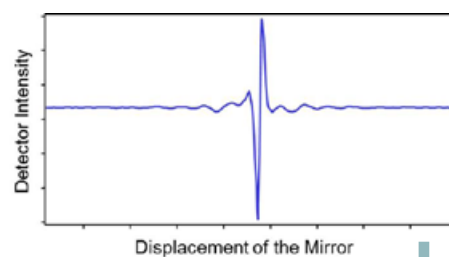
A light beam, shown in the diagram as emanating from the light source on the right side, is divided into two single beams by means of a beam splitter. One beam is focused on a fixed mirror and reflected; the other one encounters a moving mirror and is reflected as well.



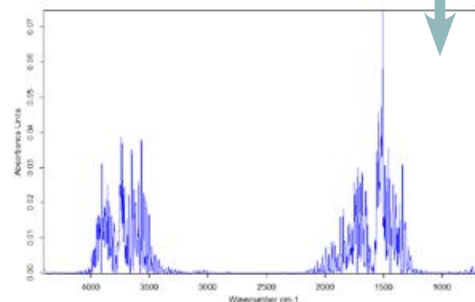
Afterwards, the two beams are merged once again and interfere – depending on the frequencies contained in the beam and the distance covered by the mirror.

The result is an interferogram characterized by a center burst and comparatively flat wings. The center burst represents the point at which the two mirrors are equidistant from the beam splitter (this means all frequencies interfere additively). Subsequently, the interferogram is transformed into a spectrum using Fourier Transformation.

Interferogram:



Spectrum:

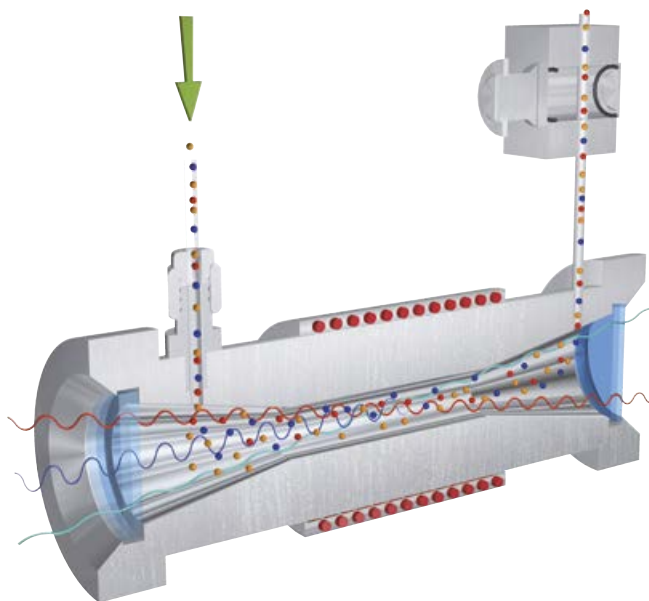


Sophisticated Gas Cell Design

To ensure highly sensitive detection of the gases evolved, it is necessary to have a gas cell with a long path length and low volume. The longer the path length, the more molecules are passing through at any given moment. On the other hand, the gas concentration is also influenced by the volume of the gas cell. The lower the volume, the higher the concentration of the gases evolved.

Developed in a collaboration between NETZSCH Analyzing & Testing and Bruker Optics, the beam-conforming metal gas cells with their optimized gas flows perfectly combine these requirements.

An additional advantage to this design is that the absence of an internal mirror prevents particle condensation, thus also preventing any influence to the detection sensitivity by a polluted mirror surface.



Characteristics of available gas cell types:

Type	Path length	Volume
Gas cell for, among others, TENSOR II* (internal or external)	123 mm	8.7 ml
Gas cell for PERSEUS® coupling (only internal)	70 mm	5.8 ml



Windows of the external gas cell for TENSOR II* with screw thread; double window technique for safe cleaning and removal

Outstanding Window Concept

The gas cell of the PERSEUS® coupling is equipped with ZnSe windows. The gas cell of the TENSOR II* uses a dual window technique with KBr windows on the outer face and ZnSe windows on the inner face. For cleaning purposes, all different window types can be easily removed and reused.

Reduced Pressure Capabilities

The spectrometers used for coupling typically work in the mid infrared range (MIR) and are operated under normal pressure. But for special tasks, a reduced pressure is also possible.



TGA/STA-FT-IR Coupling for Maximum Flexibility

All NETZSCH thermobalances (TGAs) and simultaneous thermal analyzers (STAs) are characterized by their vertical, top-loading design. This not only guarantees easy operation and sample loading, but is also in accordance with the natural gas flow path inside the furnace (warm gases have the tendency to rise) and protects the balance in an optimal way.

Because of this natural gas flow path, top-loading thermobalances are ideally suited for coupling to evolved gas analyzers such as FT-IR spectrometers, mass spectrometers and/or GC-MS systems (gas chromatograph-mass spectrometers) – and for most instruments, this can also be combined with an automatic sample changer (ASC).

Three modes of connecting FT-IR to a thermal analyzer:

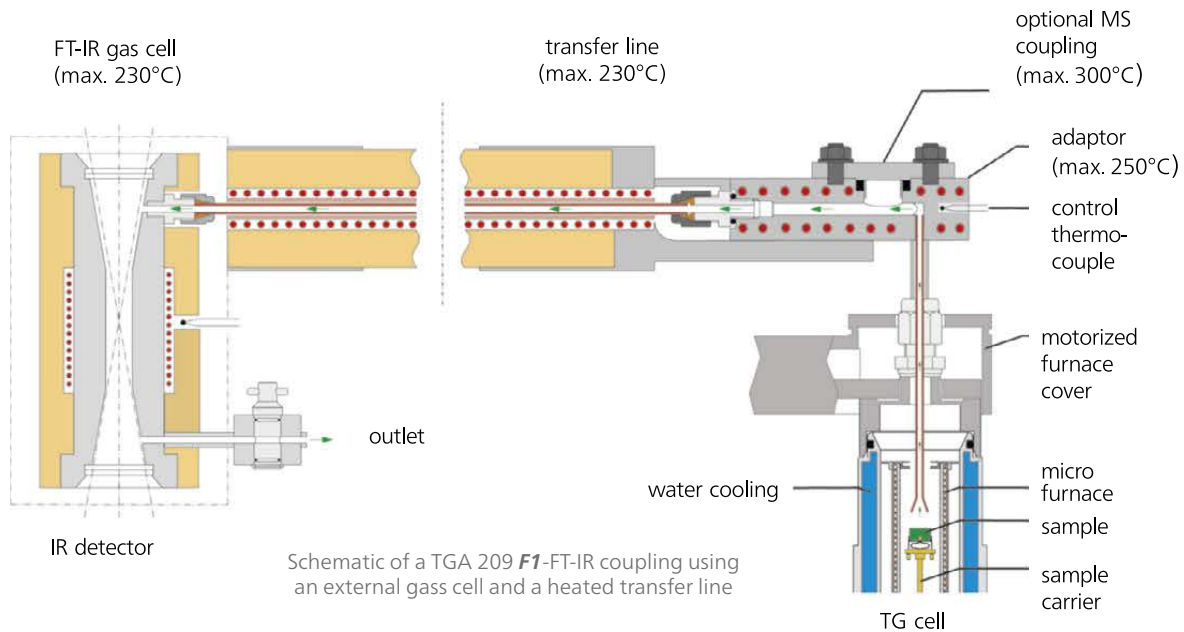
- Coupling to an external gas cell via transfer line for highest FT-IR system flexibility
- Coupling to an internal gas cell via transfer line using the FT-IR sample chamber
- Direct PERSEUS® coupling without transfer line

Defined Gas Atmosphere

Fourier Transform Infrared Spectroscopy is extremely sensitive to water and carbon dioxide. It is therefore important to establish a pure inert gas atmosphere at the sample and to minimize the H₂O and CO₂ background in the FT-IR spectra.

In order to achieve this requirement, all TGA/DSC/STA-FT-IR systems are vacuum-tight or gas-tight.





Prerequisite for Effective Coupling

An adaptor together with a short transfer line should connect the gas outlet of the TGA, DSC or STA furnace to the gas cell of the FT-IR spectrometer. The evolved gases are transferred using a carrier gas (usually nitrogen).

To prevent cold spots and thus condensation of the gases evolved, the entire gas path should be heated. The temperatures are up to 300°C for the adaptor and max. 230°C for the gas cell and transfer line.

The Right Detector for Any Application

Built-in DLaTGS (deuterated triglycine sulfate, additionally doped with L-alanine), detectors are standard for Bruker TENSOR II* and ALPHA systems. They do not require any additional cooling and are therefore particularly well-suited for TGA/DSC/STA test runs with an automatic sample changer (ASC) or for measurements of longer duration.

The external gas cells are equipped with MCT detectors (Mercury-Cadmium-Telluride) by default. They provide a significantly higher signal-to-noise ratio (compared to DLaTGS) and require liquid nitrogen cooling.



STA 2500 Regulus® with transfer line for STA-FT-IR coupling

* TENSOR II is the follow-up model of TENSOR 27

Coupling Via Transfer Line

Low-Volume Gas Path Ensures Fast Response Times



Bruker TENSOR 27 with external gas cell

TG 209 **F1** *Libra*[®]

TGA-FT-IR – Smart Gas Transfer

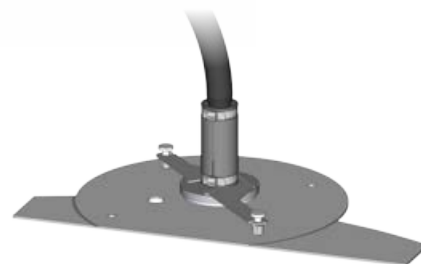
Depending on the application range, one may select from among the following available coupling possibilities:

- TG 209 **F1** *Libra*[®] coupled to an FT-IR, to FT-IR and MS (mass spectrometer), or to FT-IR and GC-MS together, via a heated adapter
- PERSEUS[®] coupling for the TG 209 **F1** *Libra*[®] (see following pages)
- TG 209 **F3** *Tarsus*[®] connected to an FT-IR transfer line directly from the furnace lid. This type of adaption is specially designed for manually operated TGA systems.

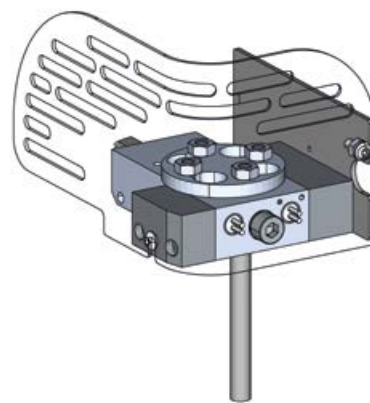
High Concentrations and Short Gas Transfer Paths for Direct Correlation with Mass Loss

The gas transfer line is characterized by a low volume and a short capillary length, thus minimizing dilution effects and guaranteeing fast response times.

The interface is optimized for Bruker FT-IR spectrometers, but is not limited to them. Please ask your NETZSCH representative for more information.



FT-IR connection for the TG 209 **F3** *Tarsus*[®]



Sophisticated heated adapter system for coupling of an FT-IR (plus MS or GC-MS) to the TG 209 **F1** *Libra*[®]

STA-FT-IR – Sensitive Evolved Gas Analysis

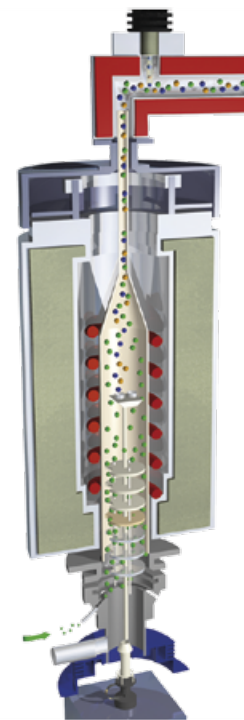
The combination of evolved gas analysis (EGA) with simultaneous thermal analysis (STA) provides also information on changes in the heat flow of the sample (DSC signal), thus allowing for an even deeper understanding of the chemical and physical processes under investigation.

Classical hyphenation of STA systems (STA 449 **F1/F3/F5** Jupiter®, STA 2500 *Regulus*®) is carried out similarly to the coupling of a TGA 209 **F1** *Libra*® with a free-standing FT-IR system (for details see previous page). The same is also valid for thermomechanical analysis systems such as TMA or dilatometers.

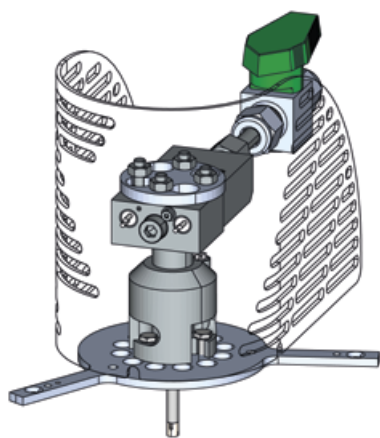
A heated adapter is the interface between the gas outlet of the STA furnace and the heated transfer line to the Bruker FT-IR spectrometer. Also here, it is possible to connect one thermal analysis instrument with two gas analyzers, such as FT-IR and QMS or FT-IR and GC-MS.

The heated adapter itself can be mounted on both sides of a double hoisting device. The temperature of the adapter is up to 300°C, the temperature of the transfer line is max. 230°C.

An external gas cell allows the operator access to a great deal of the FT-IR spectrometer's flexibility and makes it possible to add on accessories such as an ATR unit (for parallel use) or a microscope.



Schematic of the gas path of a capillary-coupled STA system



Heated adapter for capillary coupling to an STA 449 **F1/F3/F5** Jupiter® or STA 2500 *Regulus*®



PERSEUS®

Perfect Coupling of Thermal Analysis and FT-IR



PERSEUS® TG 209 F1

PERSEUS® TG 209 F1 – No Separate Transfer Line

PERSEUS® is the name given to the unique alliance between a NETZSCH thermobalance (or STA system) and a tiny but efficient FT-IR spectrometer by Bruker Optics. Its revolutionary layout sets a benchmark for state-of-the-art hyphenation.

The PERSEUS® coupling interface excels in both design and ease of handling. No separate transfer line is necessary. The built-in heated gas cell is directly connected to the gas outlet of the furnace via a heated tube. The low volume of the short gas path guarantees fast response and is quite advantageous in cases where condensable evolved gases are present. Additionally, the PERSEUS® features an extremely small footprint.

An STA 449 F1 Jupiter® system connected to a TENSOR 27 spectrometer in a side-in-side comparison with the compact PERSEUS® coupling



Bruker TENSOR 27 with external gas cell



STA 449 F1 Jupiter®

Space-Saving PERSEUS® STA 449 F1/F3

The powerful and cost-effective PERSEUS® STA 449 **F1/F3** features a compact 2-in-1 design which saves over 50% of the bench top space required for a capillary-coupled system. The short gas path with low volume provides an excellent correlation between mass losses and the gases detected.

To gain further information about the evolved gases, the PERSEUS® STA can additionally be coupled to a GC-MS system.

In order to minimize the risk of condensation, the PERSEUS® coupling interface is heated using a constant voltage. Optionally, a temperature control system is available (recommended for condensable gases). The maximum temperature of the entire gas path is set to 200°C.

Any existing STA 449 **F1/F3** system can be upgraded to the PERSEUS® configuration. Various furnaces for the temperature range from RT to 2000°C are available for this kind of coupling.



Schematic of the gas path of a PERSEUS® STA system

PERSEUS® Coupling

PERSEUS® STA 449 **F1**



No Need for Liquid Nitrogen

The DLaTGS detector (DLaTGS = L-alanine doped deuterated triglycine sulfate) employed in the spectrometer works without any cooling. The PERSEUS® system is thus perfectly suited for tests with an automatic sample changer (for 64 samples in the TG 209 **F1** or 20 in the STA 449 **F1/F3**).

The photos, depicting the instrument configuration in scale, clearly demonstrate the space-saving design of the unprecedented PERSEUS® system

DSC-FT-IR Coupling

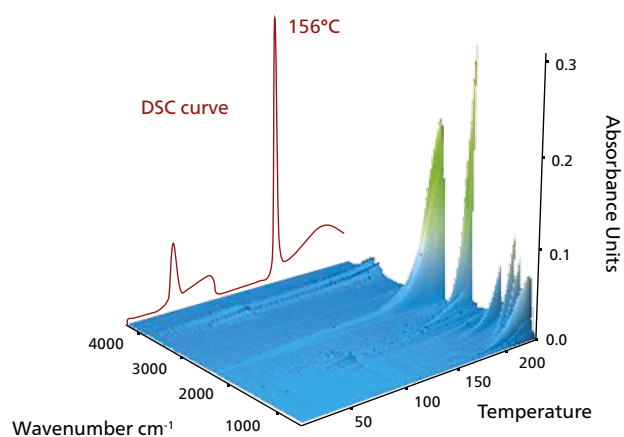
The primary objective of TGA-FT-IR experiments is to study decomposition processes. In contrast with that, the temperature profiles of DSC-FT-IR measurements are designed to avoid thermal degradation of the sample or to stay maximally at processing temperature of e.g., polymers. The main focus of DSC investigations is to analyze phase transitions such as melting and crystallization or structural changes. But even in investigations such as these, gaseous substances such as moisture or adsorbed solvents sometimes evolve. These can then be characterized using FT-IR, MS or GC-MS.

A DSC-FT-IR measurement on citric acid monohydrate ($C_6H_8O_7 \cdot x H_2O$) serves here as an example. Between 30°C and 100°C, two superimposed DSC effects are visible (red curve). According to literature, the melting of citric acid monohydrate is accompanied by dehydration – and indeed, a library search reveals the presence of crystal water.

In the temperature range from 130°C to 250°C, two additional superimposed effects occur. The endothermic one at 156°C is associated with the melting of the water-free citric acid, immediately followed by decomposition. This is reflected by a sharp increase in the intensity of the IR absorbance bands detected.



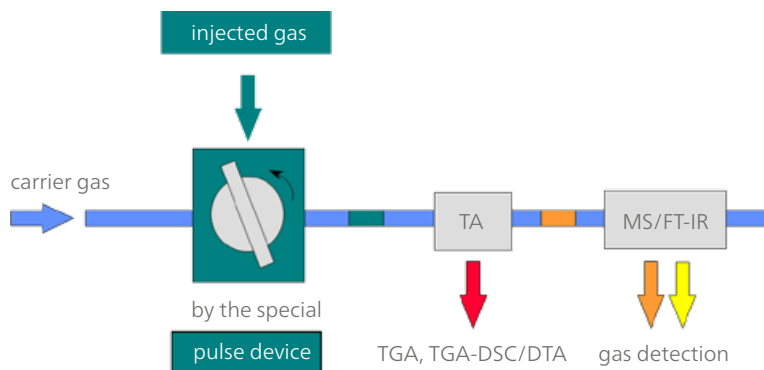
DSC 204 **F1** Phoenix® with automatic sample changer (ASC)



Measurement of 7.7 mg of citric acid monohydrate at a heating rate of 5 K/min in a nitrogen atmosphere; 3-dimensional presentation of the measured FT-IR spectra including the corresponding DSC curve in red

PulseTA[®] – A Clever Tool for Calibration, Quantification and Catalysis Studies

The idea behind the unique PulseTA[®] technique is to inject a defined amount of gas into the purge gas flow of a thermobalance (TGA) or simultaneous thermal analyzer (STA) and then monitor the corresponding changes in the sample mass, enthalpy or evolved gases.



PulseTA[®] clearly expands the scope of measurement. It can be used for three types of thermoanalytical experiments:

01 Injection of a gas which adsorbs at the sample surface

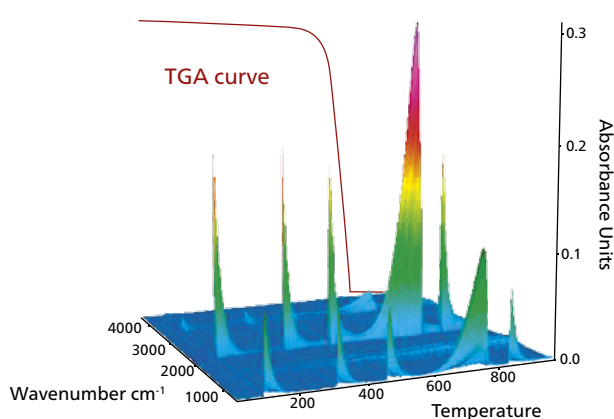
This mode offers the means to study adsorption/desorption phenomena at atmospheric pressure and at a specific temperature.

02 Injection of a gas which chemically reacts with the sample

This mode provides the opportunity to investigate all types of solid-gas reactions at incremental reaction extents (e.g., stepwise control of catalytic processes by pulsed supply of the reactive gas).

03 Injection of an inert gas

As the amount of the injected gas is known, this mode can be used for calibration of the coupled TGA/DSC/STA-FT-IR instruments for quantification purposes.



3-D plot for CO_2 calibration pulses and CaCO_3 decomposition; suitable pulses for quantification need to be repeatable and temperature-independent, and they should show a linear relationship to the injected gas concentration

Comprehensive Software

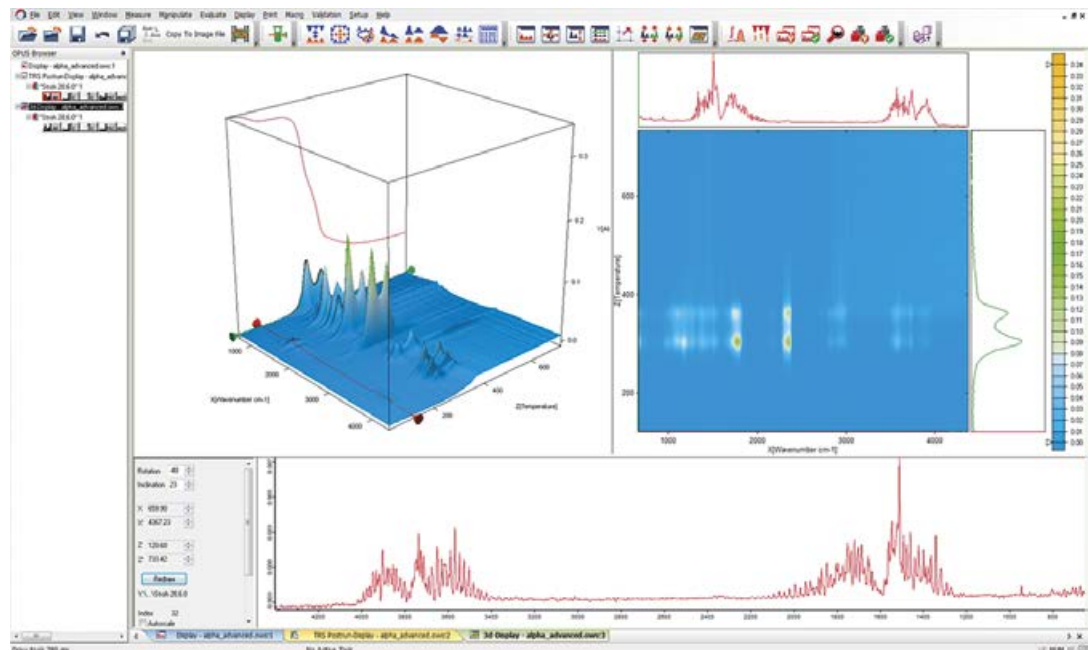
One Package for Thermal Analysis and FT-IR

Bruker OPUS and NETZSCH *Proteus*® – Unrivaled Combination

The alliance between the NETZSCH *Proteus*® software and the OPUS FT-IR software is based on effective data exchange and serves to unify the coupled system functionally.

Measurements are controlled via the NETZSCH *Proteus*® software. The user only needs to input the command for data acquisition and for the start of measurement once, and both the OPUS and *Proteus*® software will be readied with parameter inputs. Online data collection is simultaneous and synchronized to guarantee precise time and temperature correlation between all signals from the two coupled instruments during evaluation.

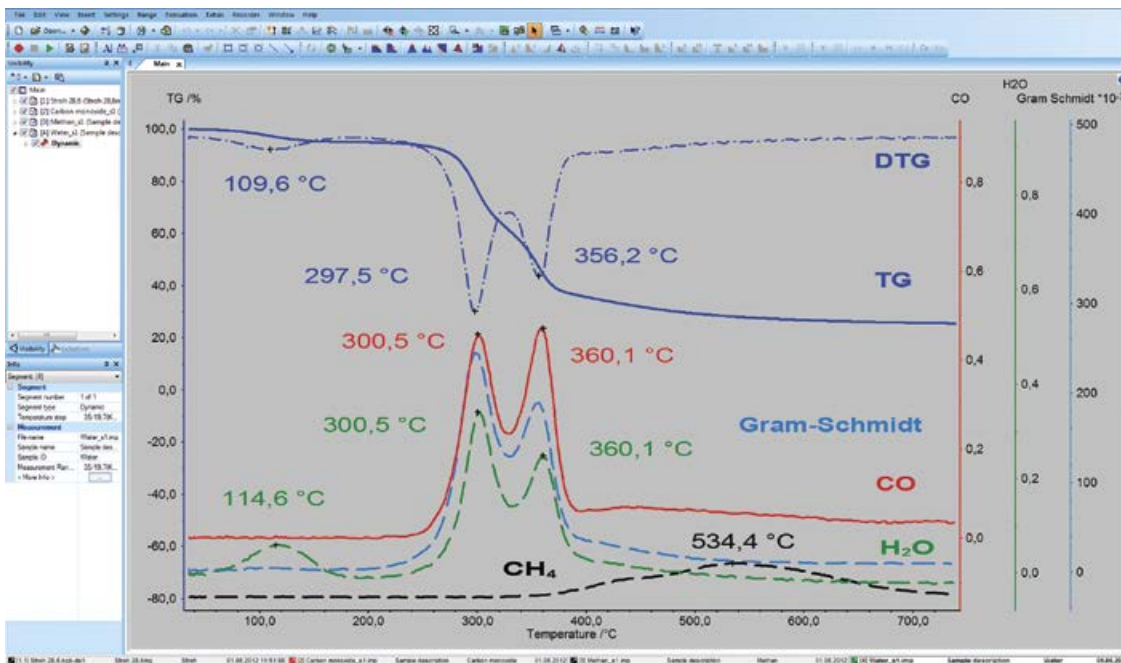
The user operates the two software packages from a single computer and has access to the full range of possibilities for data evaluation and results display in either package at any time.



Screenshot of the OPUS software during evaluation of a straw pyrolysis test: Multi-window presentation containing a 3-dimensional diagram (x-y-z view, including TGA curve and temperature information from the thermal analysis system), a 2-dimensional plot (topview on the 3-D cube) and a spectrum window, representing the spectrum at the position of the red line within the 3-D diagram

Convenient Software Setup for Maximum Ease of Use

- Full software integration – online data exchange between the two instrument software packages during the running experiment
- Simultaneous instrument control and data storage on the same computer
- Segmental activation or deactivation of the FT-IR coupling with just a few mouse clicks
- Automatic saving of data sets with identical file names (but different extensions) in the same directories
- Conjoint presentation of the Gram-Schmidt plot plus up to four pre-selected traces together with thermal analysis curves in *Proteus*® software during the experiment
- Online evaluation (SNAP SHOT) of TGA/STA/DSC measurements already including FT-IR data
- Trace calculations with evaluation of characteristic temperatures and peak areas together with TGA and DSC curves
- Combined analysis graphics of thermal analysis and FT-IR signals



Screenshot of the *Proteus*® software during evaluation of the same straw experiment as displayed before: Temperature-scaled presentation of the TGA and DTGA curves together with the Gram-Schmidt plot and the calculated traces of methane, water and carbon monoxide. A trace represents the course of the absorption intensity of a specific band as a function of time or temperature.



Advanced Materials Characterization

Fourier Transform Infrared Spectroscopy is a well-known technique in analytics. Since FT-IR spectrometers can be found in so many laboratories, it is often both convenient and logical to couple these to thermal analysis systems in order to gain a deeper understanding of the decomposition or evaporation processes under investigation. Tailored libraries support evaluation and offer quick and easy spectrum interpretation.

TGA/STA-FT-IR coupling is best suited for detecting permanent inorganic gases such as HF, CO₂ or H₂O at reasonable concentrations as well as organic molecules released from polymers, pharmaceuticals, etc.

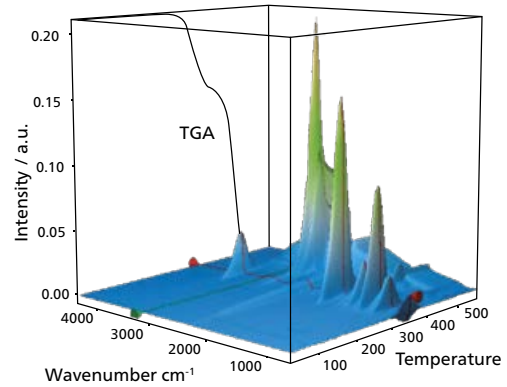
Areas of Application

- Decomposition
 - Dehydration
 - Residual solvent content
 - Pyrolysis
- Solid-gas reactions
 - Combustion
 - Oxidation
 - Corrosion
 - Catalysis
- Compositional analysis
 - Binder burn-out
 - Coal analysis
 - Polymer content
 - Ash content
- Evaporation, outgassing

Decomposition Behavior of Ethylene Vinyl Acetate (EVA)

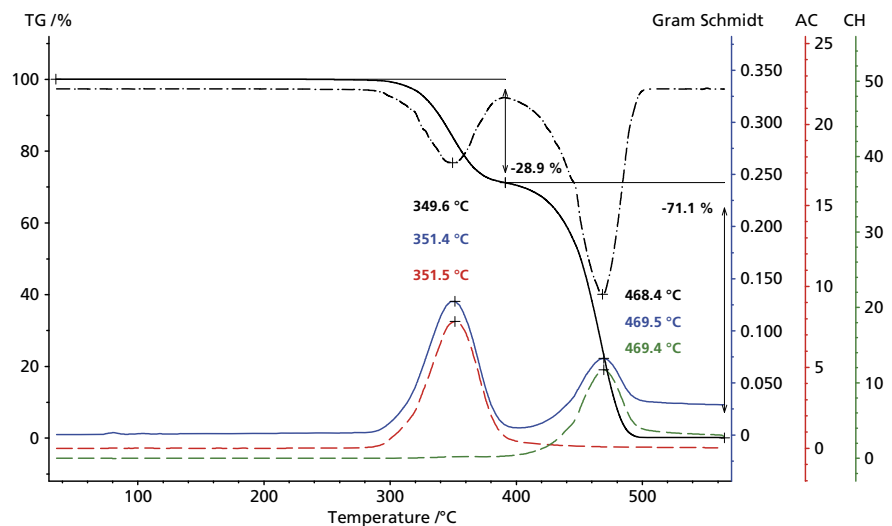
EVA is a semi-crystalline thermoplastic which is often used in the production of sport-shoe soles but is also applied in the textile industry, in agriculture and horticulture, and as a hot melt adhesive.

When heated in a nitrogen atmosphere at 10 K/min, EVA is stable up to approx. 300°C and decomposes afterwards in two steps.



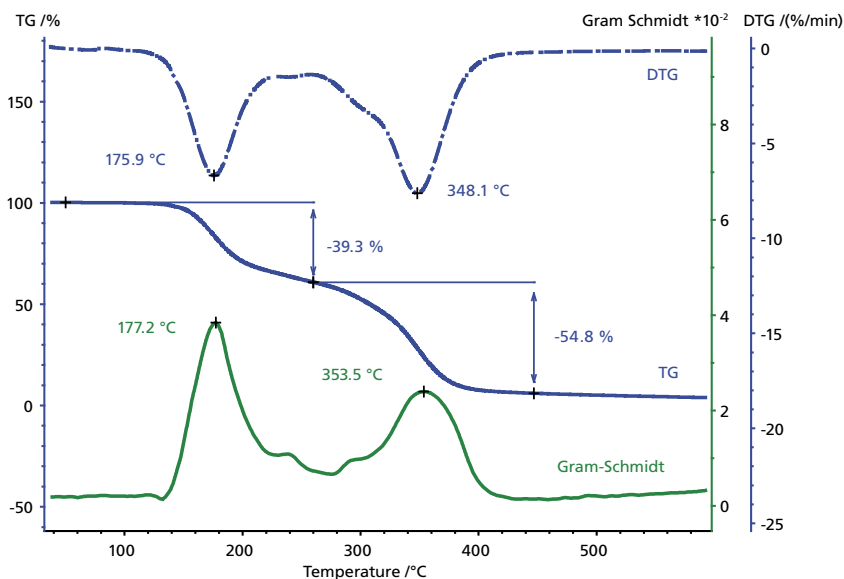
To identify the decomposition products, 2-dimensional spectra can be extracted from the 3-D cube shown above and subjected to a library search. In the present case, analysis of the FT-IR absorption intensities shows the release of acetic acid and various hydrocarbons.

Correlation with the corresponding TGA and DTGA curves (lower plot) reveals that acetic acid evolves solely in the 1st mass-loss step (at approx. 350°C), whereas the polymer backbone – illustrated by C-H vibrations – collapses within the 2nd mass-loss step (DTGA peak at 468°C) after the acetic acid has all been fully released.

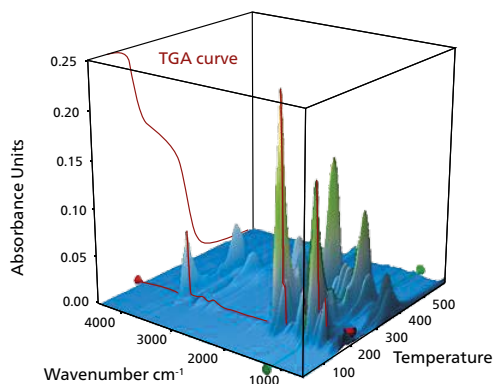


PERSEUS® TGA: Combined presentation in the *Proteus*® software of TGA (black solid) and DTGA (black dashed) curves in correlation with the Gram-Schmidt curve (blue) as well as with the individual absorption intensities of acetic acid (red) and CH (green), respectively.

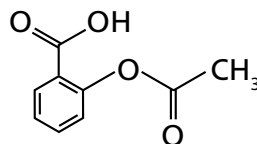
Decomposition Behavior of a Medical Drug Product: Aspirin®



TGA-FT-IR experiment on Aspirin®; sample mass: 9.14 mg, Al₂O₃ crucible, heating rate: 10 K/min, N₂ atmosphere; combined presentation of TGA curve (blue solid), DTGA curve (blue dashed) and Gram-Schmidt plot (green)



TGA-FT-IR experiment, 3-dimensional presentation of the measured FT-IR spectra and the TGA curve (red)



Stability, shelf life and residual solvents are important characteristics to study in drug substances, excipients and drug products.

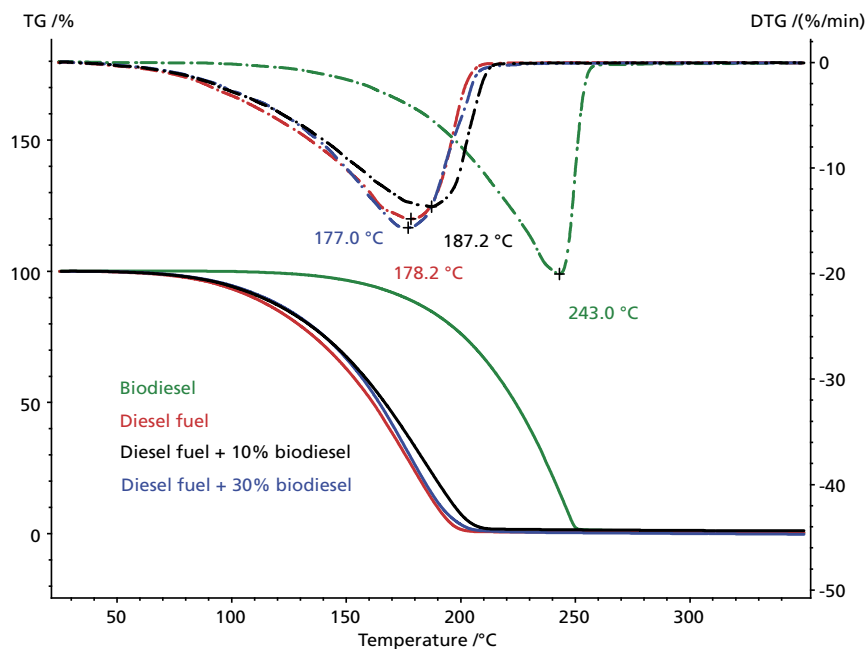
Acetylsalicylic acid, the active ingredient in Aspirin®, is responsible for its analgesic and antipyretic properties. Unfortunately, the acetyl group of this compound is very sensitive to hydrolysis and even reacts with humidity. Adding excipients and coating the tablets with paraffin are possible measures to suppress this reaction.

For the TGA-FT-IR investigation shown here, a piece of an Aspirin® tablet was heated up to complete decomposition, leading to two main mass-loss steps (according to the TGA and DTGA profiles). The FT-IR analysis of the gas phase above the sample yields acetic acid, salicylic acid, phenol and carbon dioxide as key components. This result corresponds well with the reaction and decomposition scheme of acetylsalicylic acid which can be found in literature. The high boiling components are efficiently transferred through the heated transfer line to the gas cell and clearly detected by FT-IR.

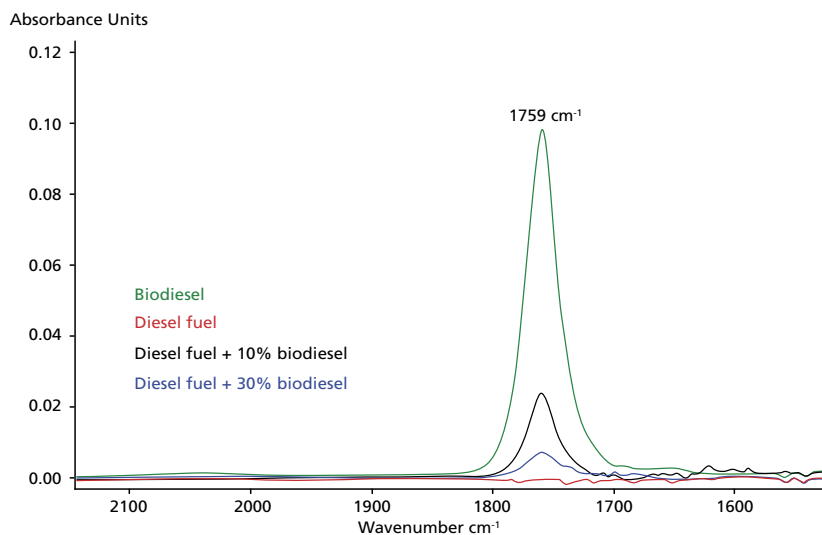
The boiling point of salicylic acid is specified as 211°C and that of phenol as 181°C, both at a surrounding pressure of 1013 mbar.



Differentiation between Diesel Fuel and Biodiesel



TGA comparison of various diesel fuel/biodiesel mixtures; sample masses: 10 to 11.5 mg, Al₂O₃ crucibles, heating rate: 10 K/min, N₂ atmosphere



Comparison of the IR spectra of all measured samples. Zoom in the region between 1500 and 2200 cm⁻¹.

For some years, fuel has been mixed with a certain amount of bio fuel. This also applies to diesel. In Europe, biodiesel is mostly rapeseed methyl ester; in the US, the source is almost exclusively soybean oil.

Various norms define the chemical composition, inorganic content, density and stability, etc. In order to investigate the decomposition behavior and thermal stability, pure diesel fuel, pure biodiesel and two mixtures (10% and 30% biodiesel) were measured here using TGA-FT-IR.

There is a clear difference in the thermal stability between the vaporization temperatures of pure biodiesel (green) and pure diesel fuel (red) as discovered by TGA, but there is no apparent systematic relationship with regard to the mixtures. The comparison of the corresponding IR spectra, however, reveals a band at 1759 cm⁻¹ which is specific for biodiesel. It is most probably related to the ester group. The intensity of this band even shows a relationship with the biodiesel proportion: it increases with increasing biodiesel content.

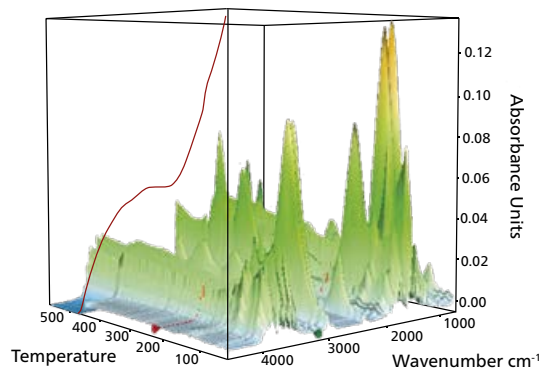


Simulation of Application and



Curing of Water-Based Paint

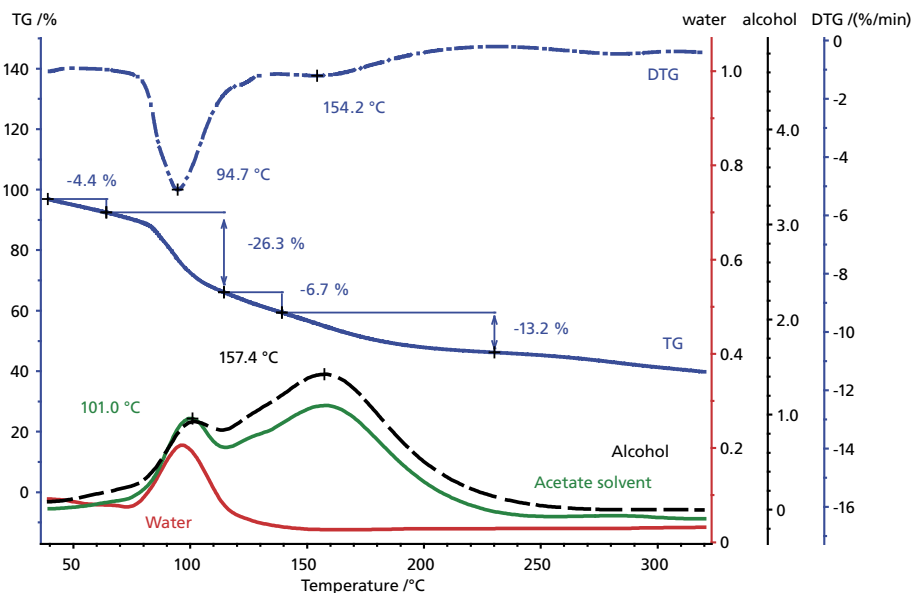
Solvent-based products typically contain high levels of "Volatile Organic Compounds" (VOCs), associated with relatively long drying times and a strong smell. Water-based products usually have much lower VOC levels. However, volatile components in water-based paints can still be an environmental issue – e.g., during application if by-products of the curing reaction evolve.



TGA-FT-IR experiment on a hydro clear coat; 3-dimensional presentation of the measured FT-IR spectra and TGA curve (brown) spectra

31.9 mg of a two-component hydro clear coat were analyzed with the TGA-FT-IR coupling. The sample was heated to 300°C at a rate of 5 K/min in a nitrogen flow of 45 ml/min.

As the measurement graphs show, one of the main weight-loss occurrences prior to 250°C is clearly attributed to water, but a significant contribution also comes from hydrocarbons such as alkyl acetates and aliphatic alcohols. The maximum evolution rate for these latter components is shown by the peaks in the traces (temperature-dependent intensities) at 157°C. This means that no indication of harmful or toxic volatiles is found during drying of this clear coat.



Drying and curing of a water-based clear coat; sample mass: 31.9 mg, Al_2O_3 crucibles; combined presentation of TGA curve (blue solid), DTGA curve (blue dashed) and traces of water (red), alcohol (black) and alkyl acetates (green)

Manufacturing Conditions

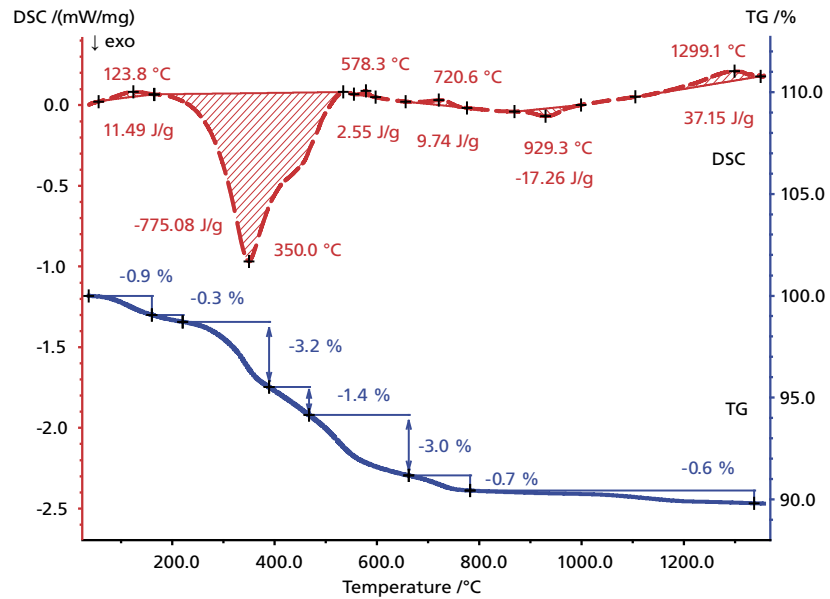
Firing of Clay

To save energy in buildings, wall constructions should have low thermal conductivity. One way to achieve this is to use highly porous building bricks. Various organic products capable of generating a high volume of voids are mixed into the clay to form cavities during firing.

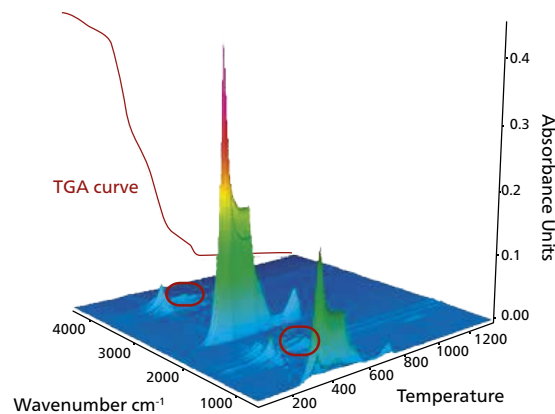
In this example, 107.6 mg of a clay green body was heated at a rate of 10 K/min in Pt/Rh crucibles in flowing air (50 ml/min). The predominant effect during heating is the burnout of the organics between 200°C and 550°C, which is accompanied by a high energy release (775 J/g).

The main volatiles emerging during this process are water and carbon dioxide, but the FT-IR also clearly detects the evolution of HF (around 4000 cm⁻¹) and SO₂ (around 1300 to 1400 cm⁻¹) from the clay (red circles).

Identification of such emissions allows for optimization of the firing process from both economical and ecological standpoints.



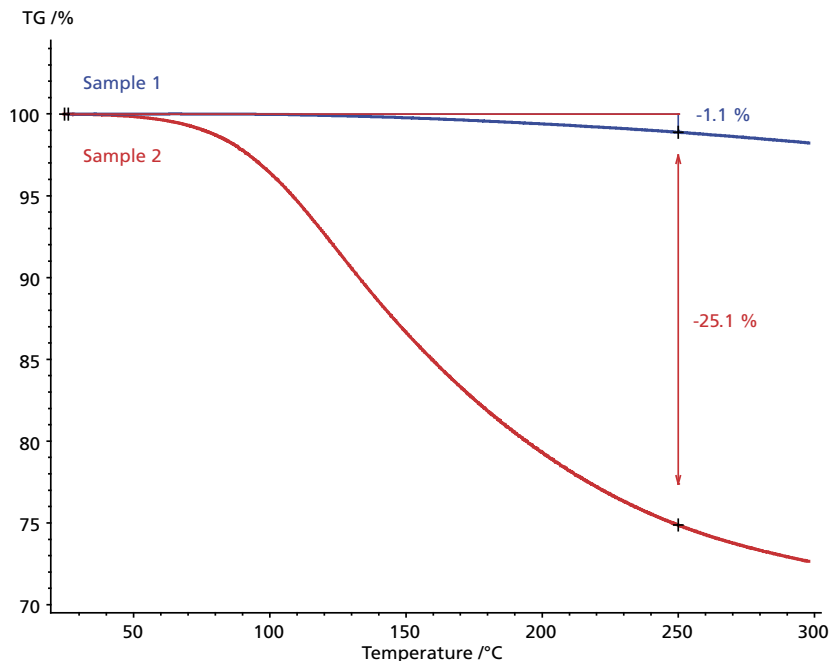
Mass changes and energetic changes of a clay for porous bricks; presentation of the TGA curve (blue) and the corresponding DSC curve (red)



STA-FT-IR experiment on a clay, 3-dimensional presentation of the measured FT-IR spectra, including TGA curves from the STA



Process Optimization



Comparison of the TGA results of two TGA-FT-IR experiments; sample masses: 24.5 mg and 26.3 mg, Al₂O₃ crucibles, heating rate: 10 K/min, N₂ atmosphere

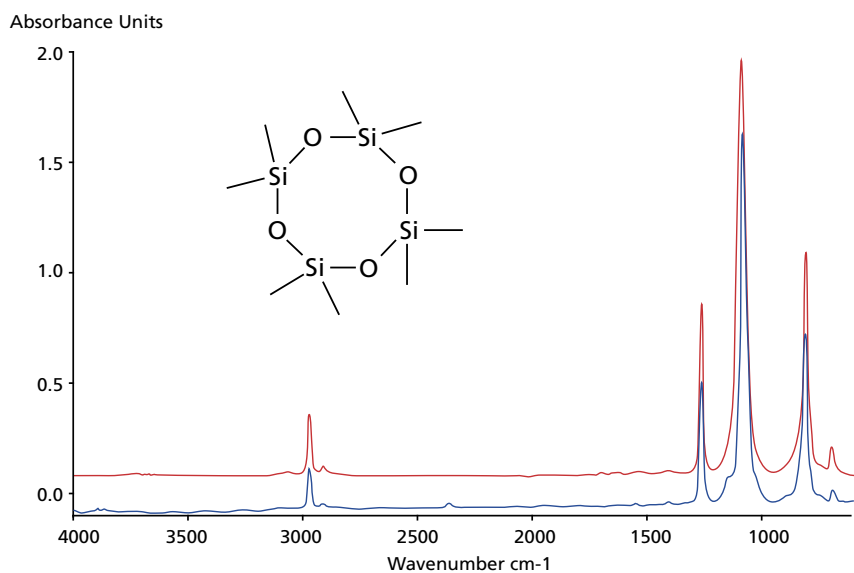
Manufacturing of Silicones

TGA-FT-IR coupling can be of great help in detecting causes of failure during polymer processing. Here, two control samples of silicones were tested – one of them (sample 2) revealed manufacturing problems.

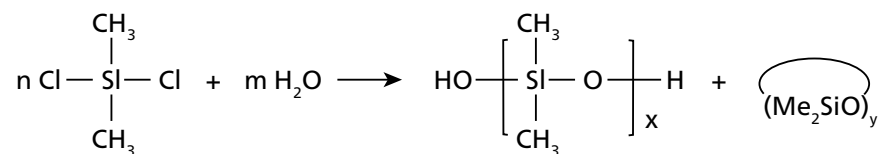
The TGA curves exhibit significantly different thermal behavior. Sample 2 loses approx. 25% of its weight whereas the mass loss for sample 1 is only about 1% up to 250°C.

To identify the gases evolved, a single spectrum was extracted at the point of maximum IR intensity and maximum mass-loss rate (about 126°C; DTGA curve not demonstrated here). Library comparison suggests cyclooctamethyltetrasiloxane with a perfect agreement in band pattern with the experimental spectrum.

The polymerization mechanism for the generation of silicones often proceeds via cyclosiloxanes as intermediate products (presented below in a simplified manner). Organochlorosilanes such as dimethylchlorosilane (CH₃)₂SiCl₂ are hydrolyzed and form silanoles which react at higher temperatures and in the presence of catalysts to become the desired end product. In the present case, neither water nor the educt dimethylchlorosilane contributes to the observed mass loss. This leads to the conclusion that in the case of sample 2, the reaction was partially halted at the intermediate product.



Comparison of the measured spectrum extracted at 126°C and the results of the library search (cyclooctamethyltetrasiloxane)



Technical Specification

Characteristic Data for All Available FT-IR Coupling Systems


Coupling system	TG 209 F1 <i>Libra</i> ®; TG 209 F3 <i>Tarsus</i> ®	DSC 404 F1/F3 ; STA 2500 <i>Regulus</i> ®; STA 449 F1/F3/F5	PERSEUS® STA 449 F1/F3 ; PERSEUS® TG 209 F1	TMA 402 F1/F3 ; DIL 402 <i>Expedis</i> <i>Select/Supreme</i>	DSC 204 F1 <i>Phoenix</i> ®
Temperature range ⁴	RT-1000°C (F3) 10°C(RT)-1100°C (F1)	RT-1600°C (<i>Regulus</i> ®, F5) -150°C-2000°C (F1/F3)	RT-2000°C (STA) RT-1100°C (TGA)	RT-1550°C (TMA) RT-2000°C (DIL)	RT-700°C
Measurements under reduced pressure (for solvent separation)	Yes (F1)	Yes	Yes	Yes	No, atmospheric pressure only
Vacuum-tightness of the complete system	Yes (F1)	Yes	Yes	Yes	No
Additional automatic sample changer operation ¹	Yes (F1)	Yes (DSC 404, STA 449) No (<i>Regulus</i> ®)	Yes	No	Yes
Possible Bruker FT-IR spectrometer types ²	TENSOR, VERTEX	TENSOR, VERTEX	ALPHA	TENSOR, VERTEX	TENSOR, VERTEX
Stand-alone operation of the FT-IR	Yes	Yes	No	Yes	Yes
Additional analytical instruments to be coupled	MS or GC-MS (F1)	MS or GC-MS	MS or GC-MS	MS or GC-MS	MS or GC-MS
T _{max} transfer line, transfer tube/ adapter head	230°C/300°C	230°C/300°C	200°C	230°C/300°C	230°C/300°C
Set-up gas cell	Internal or external	Internal or external	Internal	Internal or external	Internal or external
Material gas cell	Stainless steel	Stainless steel	Nickel-coated aluminum	Stainless steel	Stainless steel
Gas cell – path length	123 mm	123 mm	70 mm	123 mm	123 mm
Gas cell – volume	8.7 ml	8.7 ml	5.8 ml	7.8 ml	8.7 ml
Detector ³	DLaTGS (internal) or MCT (external)	DLaTGS (internal) or MCT (external)	DLaTGS (internal)	DLaTGS (internal) or MCT (external)	DLaTGS (internal) or MCT (external)

1 Larger Dewar recommended for MCT detector

2 For coupling with FT-IR spectrometers from other suppliers, please ask your NETZSCH representative

3 MCT detector requires LN₂ cooling

4 For most instruments, variable furnaces are required to cover the stated temperature range



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