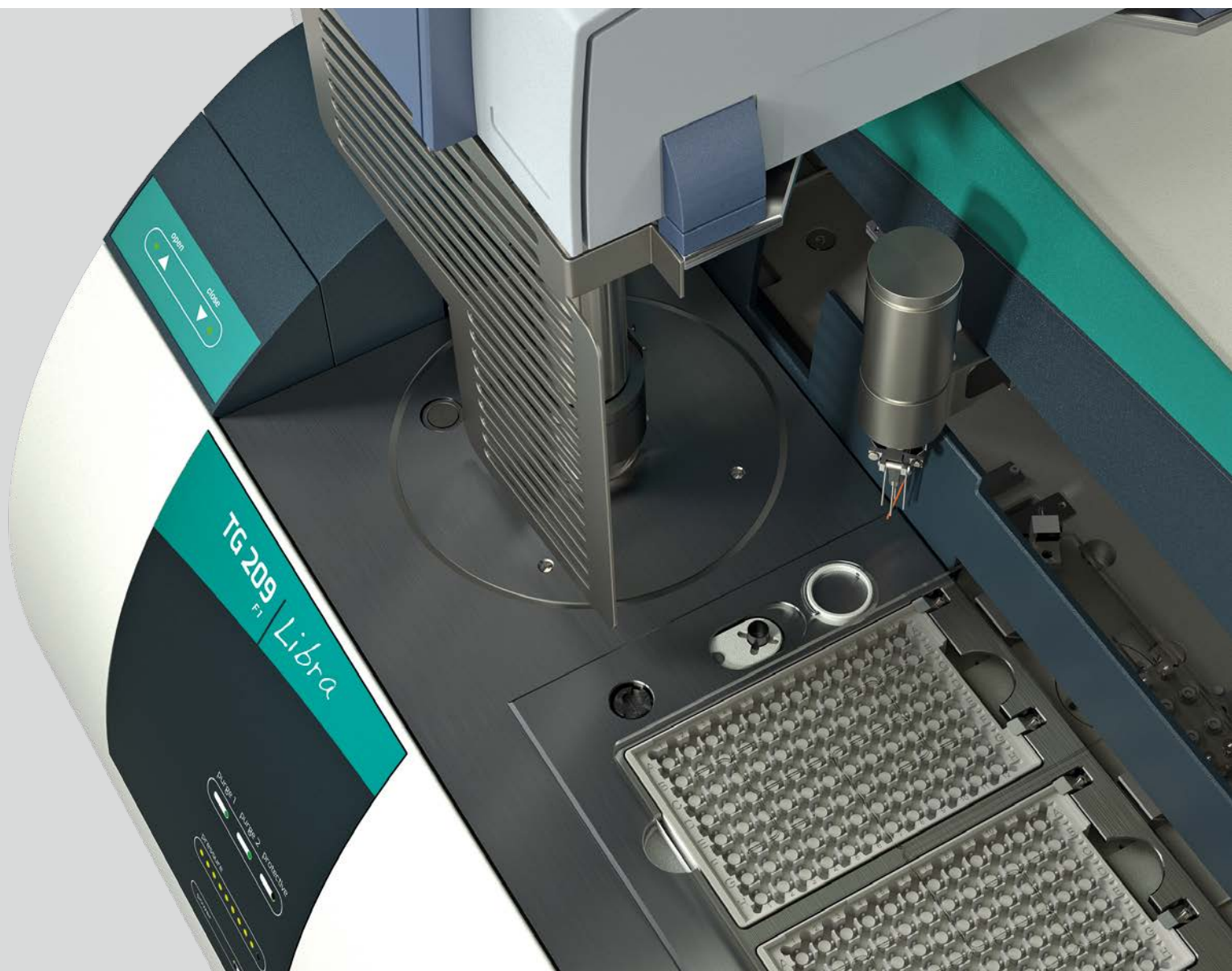


**NETZSCH**

Proven Excellence.



## Fourier Transform Infrared (FT-IR) Spectrometers Coupled to Thermal Analysis

Concepts, Instruments and Applications from RT to 2000°C

Analyzing & Testing

# Thermal Analysis Plus FT-IR

More Than Just the Sum of Its Parts

A hand in a white lab coat points towards a white rounded rectangle containing the text "FT-IR-Coupling". The background is a blurred image of a person in a lab coat with several semi-transparent blue rounded rectangles overlaid on it.

FT-IR-  
Coupling

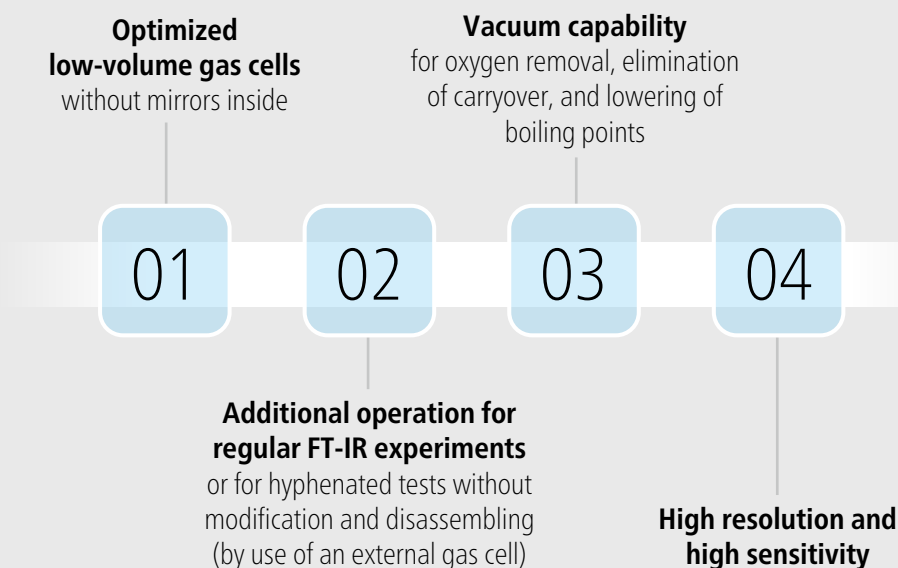
## Perfect Combination Bridges the Analytical Gap and Facilitates Fingerprint Analysis

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*<sup>®</sup> software for thermal analysis and the OPUS software for the FT-IR measurement 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

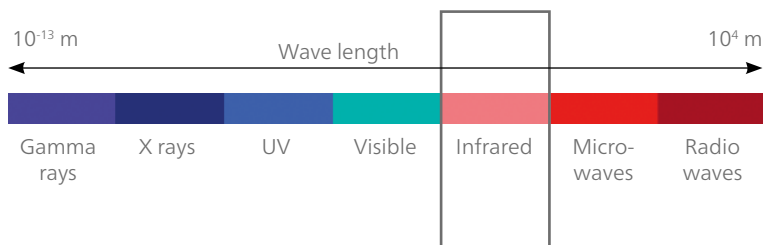


### Great Variety of Configurations to Meet all Needs

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

\* not with ASC

# Optimized TG/STA/DSC – FT-IR Coupling



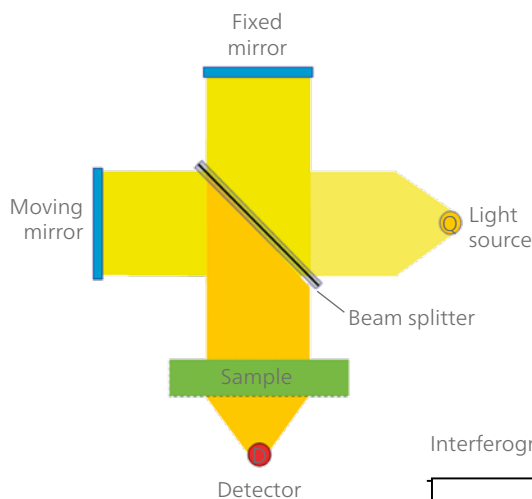
**Infrared spectroscopy** is a classical technique which depends on the interaction of infrared radiation with the vibrating dipole moments of molecules. It provides a characteristic spectrum for each substance, with the exception of homonuclear diatomic molecules or noble gases. Examples of homonuclear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

## Working principle of a 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.

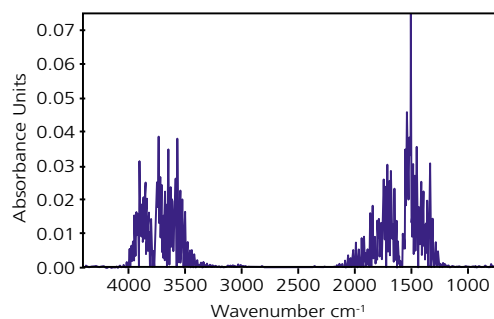
The result is an interferogram characterized by a center burst and comparatively flat wings. The center burst represents the point at which the two mirrors are equidistant from the beam splitter (this means all frequencies interfere additively). Subsequently, the interferogram is transformed into a spectrum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



Interferogram:



Spectrum:

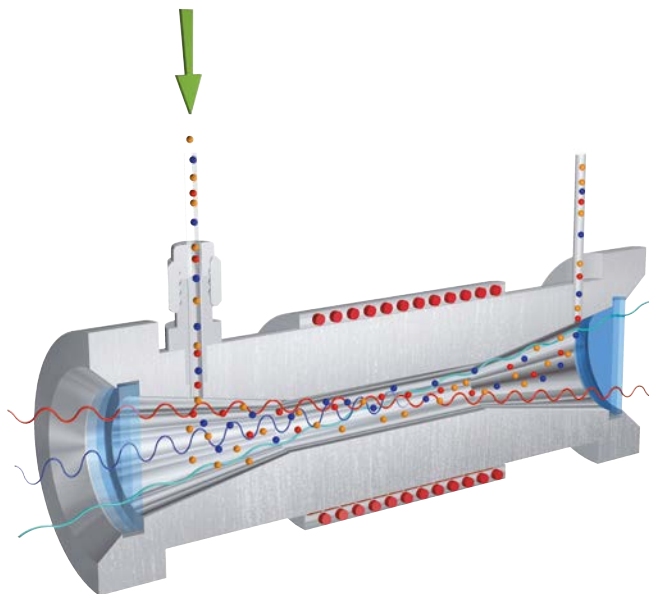


## Sophisticated Gas Cell Design

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 interacting with the IR beam. 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.



Design of the gas cell for optimized gas flows

## Characteristics of available gas cell types:

Type	Gas cell for, among others, INVENIO (internal or external)	Gas cell for PERSEUS® coupling (only internal)
Length	123 mm	70 mm
Volume	11.8 ml	5.8 ml
Windows	KBr windows	ZnSe windows
Windows removal for cleaning purpose	✓	✓

## 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.



Windows of the external gas cell for the INVENIO spectrometer for safe cleaning and removal

# TGA/STA-FT-IR Coupling for Maximum Flexibility



Bruker INVENIO with external gas cell coupled to the NETZSCH TG 209 *F1 Libra*<sup>®</sup> with ASC and QMS 403 *Aëolos Quadro*

All NETZSCH thermobalances 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 underneath 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 compartment
- Direct PERSEUS<sup>®</sup> coupling without transfer line

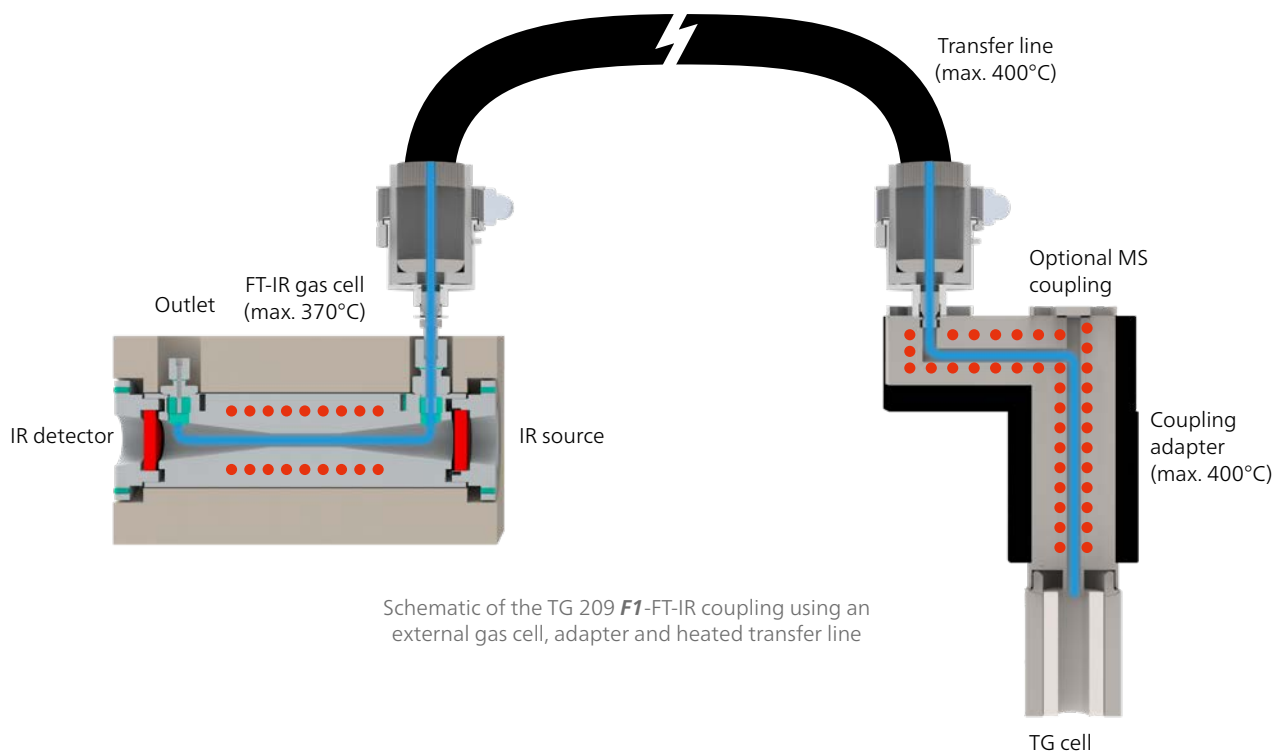
## Defined Gas Atmosphere

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 H<sub>2</sub>O and CO<sub>2</sub> 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.



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



### Prerequisite for Effective Coupling

An adapter 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 400°C for the adapter and max. 400°C for transfer line.

### The Right Detector for Any Application

Built-in DLaTGS (deuterated triglycine sulfate, additionally doped with L-alanine), detectors are standard for Bruker INVENIO 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 DLaTGS detectors by default but is also available with MCT (Mercury Cadmium Telluride). MCT detectors provide a significantly higher signal-to-noise ratio (compared to DLaTGS) and require liquid nitrogen cooling.

### Advantages of the NETZSCH-Bruker Coupling at a Glance

- Short transfer path
- Continuously heated interface and gas cell
- Minimized risk of condensation
- Mirror-free gas cell
- Optimized volume/pathlength ratio
- Tests with automatic sample changer for high sample throughput and great variability of measurement parameters

# Coupling Via Transfer Line

Low-Volume Gas Path Ensures Fast Response Times



Bruker INVENIO with external gas cell and an ATR unit in the FT-IR sample compartment



TG 209 **F1** Libra®

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

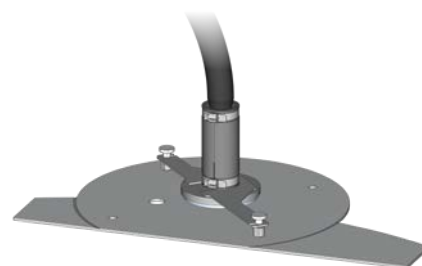
## TGA-FT-IR – Smart Gas Transfer

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

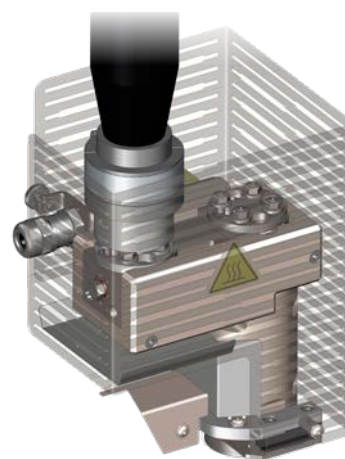
The interface is optimized for Bruker FT-IR spectrometers, but is not limited to them. Please ask your NETZSCH representative for more information.

## External gas cell – Free Internal FT-IR Compartment

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 or a microscope.



FT-IR connection for the TG 209 **F3**



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



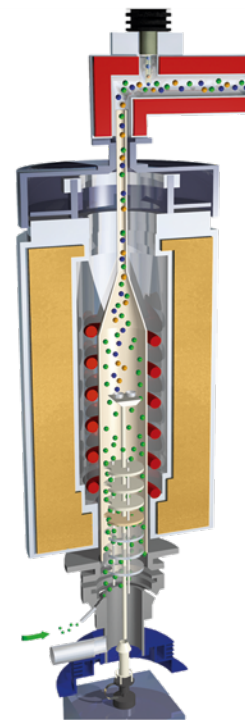
## STA-FT-IR – Sensitive Evolved Gas Analysis

The combination of evolved gas analysis (EGA) and 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.

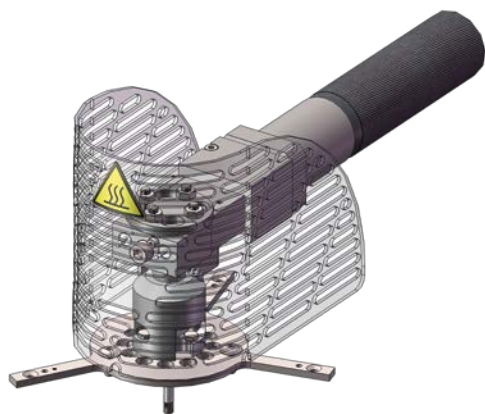
Classical hyphenation of STA systems (STA 449 **F1/F3/F5** *Jupiter*®, STA 449 **F3** *Nevio*, STA 2500 *Regulus*) is carried out similarly to the coupling of a TG 209 **F1** *Libra*® or a TG 209 **F1** *Nevio* with a free-standing FT-IR system (for details see previous page). The same is also valid for thermo-mechanical 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 the STA's double hoisting device. The temperature of the adapter is up to 300°C, the temperature of the transfer line is max. 230°C.



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



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



# PERSEUS®

## Perfect Coupling of Thermal Analysis and FT-IR



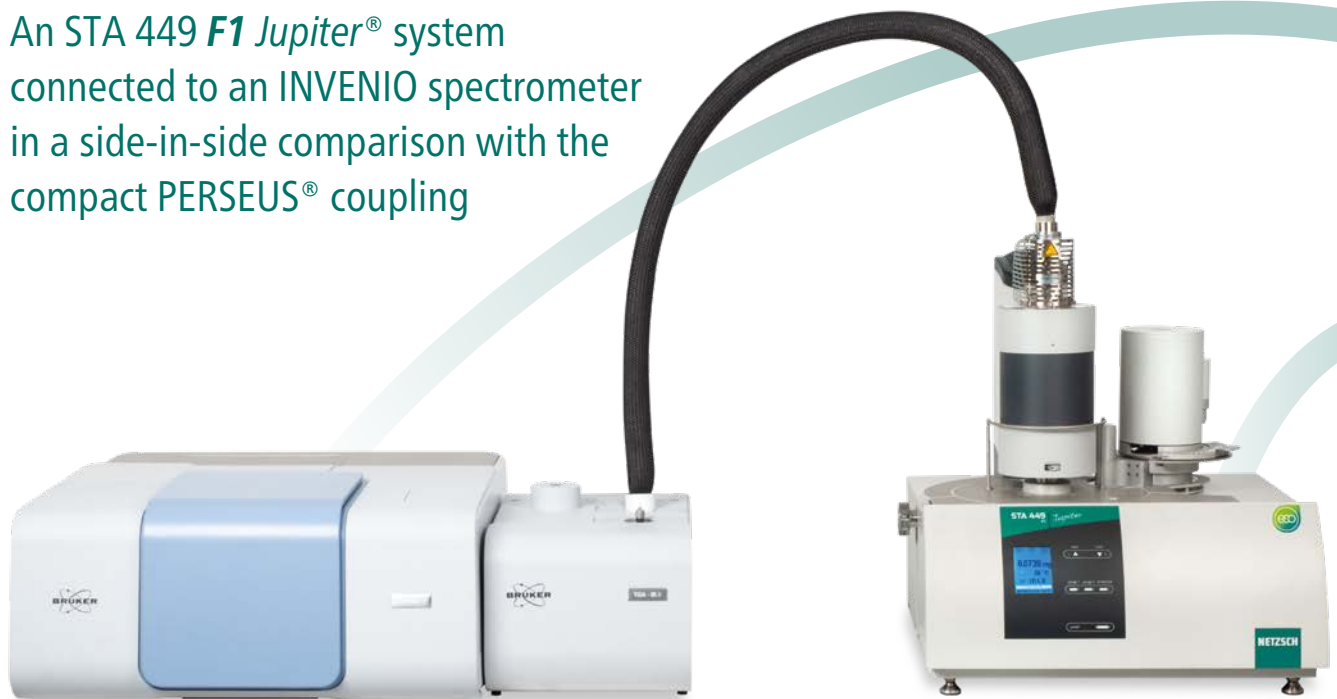
PERSEUS® TG 209 **F1** Libra®

### 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 an INVENIO spectrometer in a side-in-side comparison with the compact PERSEUS® coupling



Bruker INVENIO with external gas cell

STA 449 **F1** Jupiter®

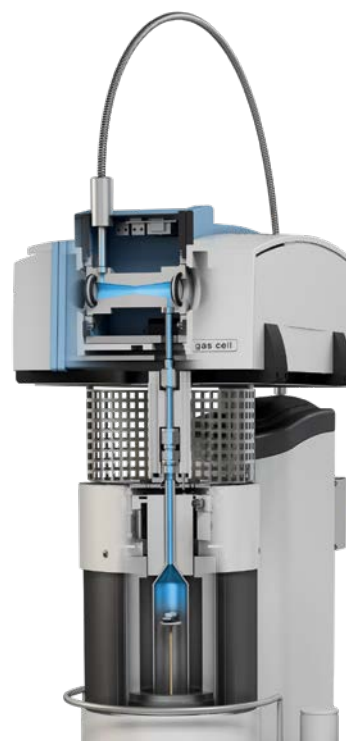
## Space-Saving PERSEUS® STA 449

The powerful and cost-effective PERSEUS® STA 449 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.

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 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.

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



Schematic of the gas path of a PERSEUS® STA system

## PERSEUS® Coupling

PERSEUS® STA 449 **F1** Jupiter®



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

## 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 up to 204 samples in the TG 209 **F1** or 20 in the STA 449).

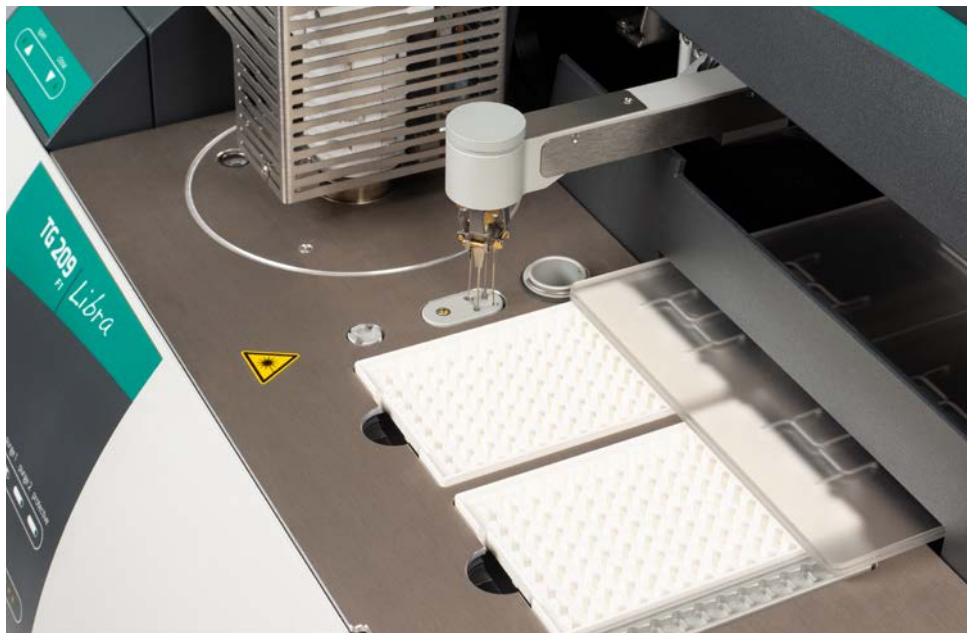
# DSC-FT-IR Coupling

Usually, TGA-FT-IR experiments study decomposition processes. In contrast, 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.

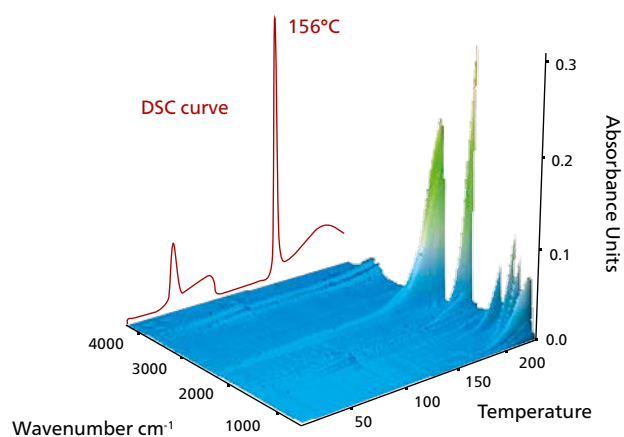
The main focus of DSC investigations is to analyze phase transitions such as melting and crystallization or structural changes. However, gaseous substances such as moisture or adsorbed solvents sometimes evolve. These can then be characterized using FT-IR, MS or GC-MS.

The plot shows a DSC-FT-IR measurement on citric acid monohydrate ( $C_6H_8O_7 \cdot x H_2O$ ). 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.

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



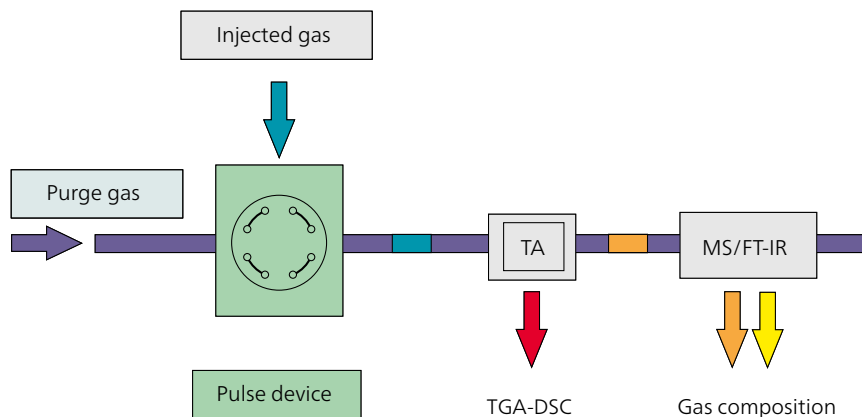
TG 209 **F1** Libra® with automatic sample changer (ASC)



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

# PulseTA<sup>®</sup> – A Clever Tool for Calibration, Quantification and Catalysis Studies

PulseTA<sup>®</sup> technique is used 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 therewith related changes in the sample mass, enthalpy or changes in the evolved gases.



PulseTA<sup>®</sup> allows for three types of thermoanalytical experiments:

## 01 Injection of a gas which adsorbs at the sample surface

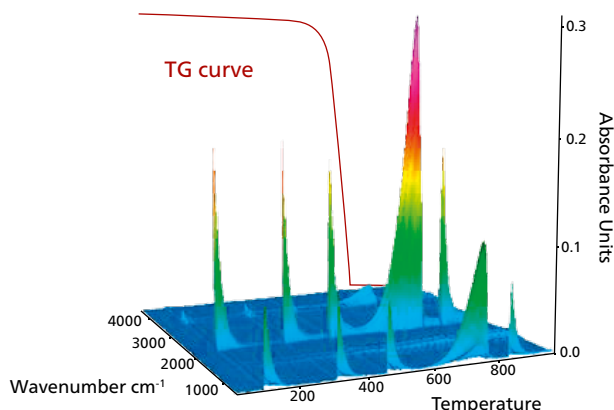
This mode offers the means to study adsorption/desorption phenomena at atmospheric pressure and at a specific temperature.

## 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.



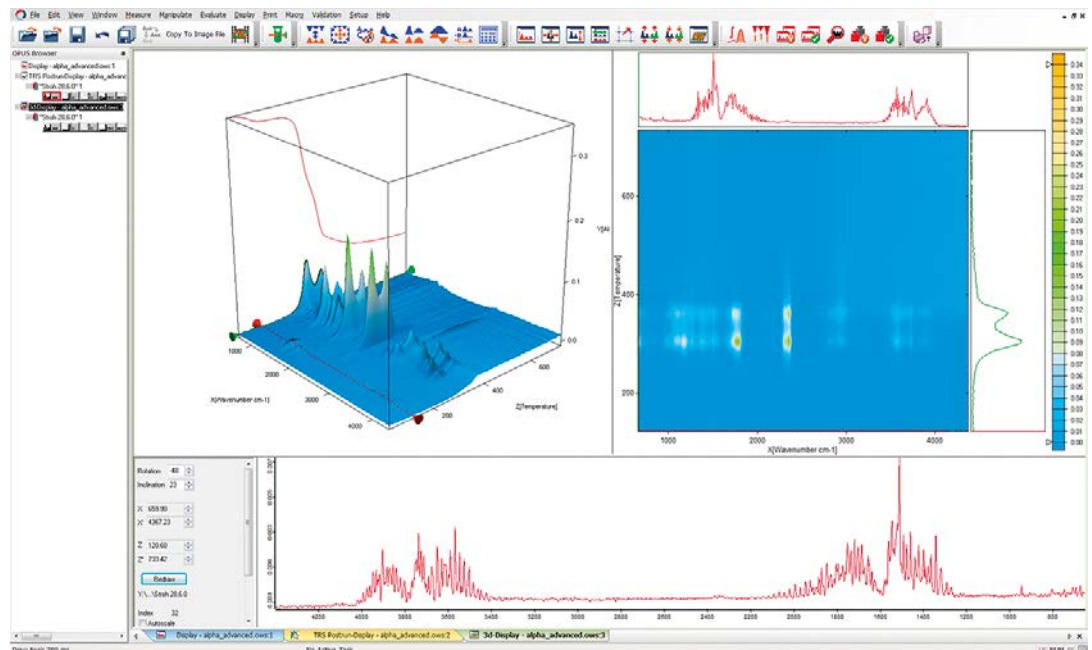
# Comprehensive Software

## Bruker OPUS and NETZSCH *Proteus*® – Unrivaled Combination for Maximum Ease of Use

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.

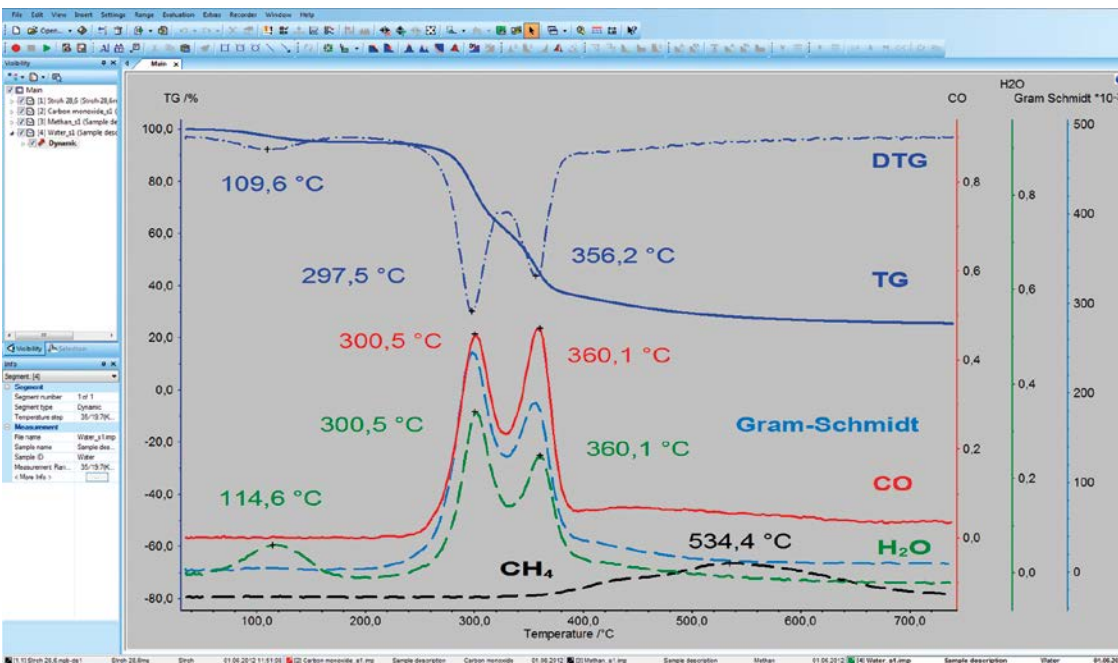
Measurements are controlled via the NETZSCH *Proteus*® software. The user only needs to input the command for data acquisition and for the start of measurement once, and both the OPUS and *Proteus*® software will be readied with parameter inputs. Online data collection is simultaneous and synchronized to ensure 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-D diagram (x-y-z view, including TGA curve and temperature information from the thermal analysis system), a 2-D 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

- Full software integration – online data exchange between the two instrument software packages during the running experiment
- Simultaneous instrument control and start-stop function
- Segmental activation or deactivation of the FT-IR coupling with one mouse click
- Automatic saving of data sets for both measurements (TGA and FT-IR) with identical file names (but different extensions) in the same directories
- Measurements with automatic sample changer allow for individual FT-IR measurement parameter for each position
- 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 during the measurement
- 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
- Multi-component search in OPUS
- Identification by various gas phase libraries, e.g., TGA-FT-IR library of polymers by NETZSCH



Screenshot of the *Proteus*® software during evaluation of the same straw experiment: Temperature-scaled plot of the TGA and DTG curves together with the Gram-Schmidt plot and the calculated traces of methane, water and carbon monoxide (course of the absorption intensity of a specific band)



# 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, CO<sub>2</sub> or H<sub>2</sub>O at reasonable concentrations as well as organic molecules released from polymers, pharmaceuticals, etc.

## Areas of Application

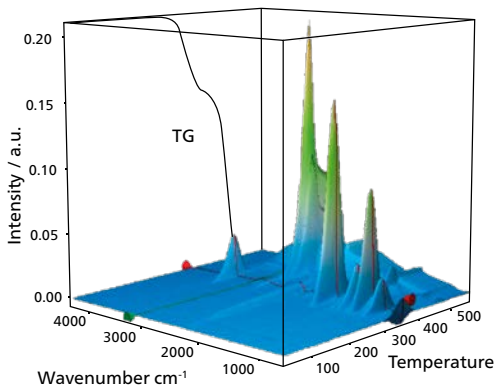
- Decomposition
  - Dehydration
  - Residual solvent content
  - Pyrolysis
- Solid-gas reactions
  - Combustion
  - Oxidation
  - Corrosion
  - Catalysis
- Compositional analysis
  - Binder burn-out
  - Coal analysis
  - Polymer content
  - Ash content
- Evaporation
- Outgassing



## Decomposition Behavior of Ethylene Vinyl Acetate (EVA)

EVA is a semi-crystalline thermo-plastic 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.

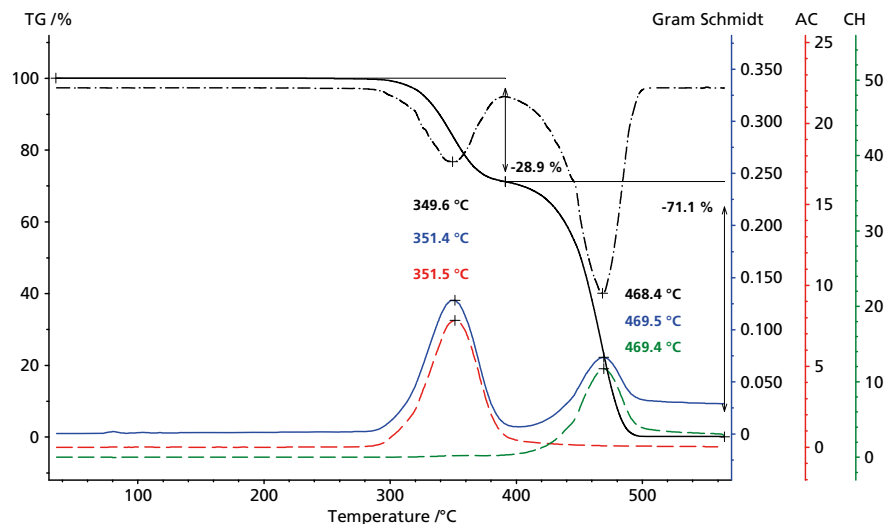
When heated in a nitrogen atmosphere at 10 K/min, EVA is stable up to approx. 300°C and decomposes afterwards in two steps.



TG-FT-IR experiment, 3D presentation of the measured FT-IR spectra and the TGA curve

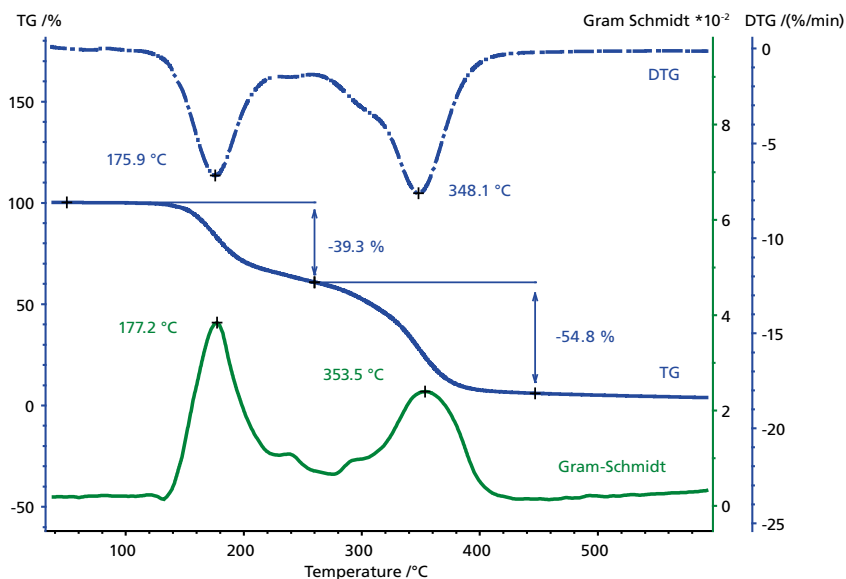
To identify the decomposition products, 2-D spectra can be extracted from the 3-D cube shown above and subjected to a library search. In the present case, analysis of the FT-IR absorption intensities shows the release of acetic acid and various hydrocarbons.

Correlation with the corresponding TGA curves (lower plot) reveals that acetic acid evolves solely in the 1<sup>st</sup> mass-loss step (at approx. 350°C), whereas the polymer backbone – illustrated by C-H vibrations – collapses within the 2<sup>nd</sup> mass-loss step (DTG 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 DTG (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®

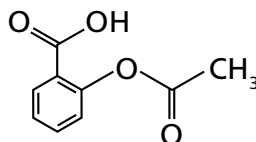


TGA-FT-IR experiment on Aspirin®; sample mass: 9.14 mg, Al<sub>2</sub>O<sub>3</sub> crucible, heating rate: 10 K/min, N<sub>2</sub> atmosphere; combined presentation of TG curve (blue solid), DTG curve (blue dashed) and Gram-Schmidt plot (green)

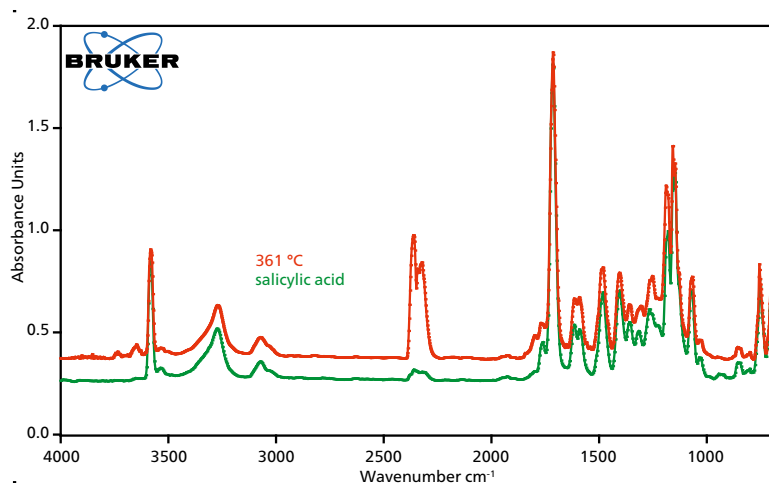
Acetylsalicylic acid is the active ingredient in Aspirin®. In a humid environment, the acetyl group of this compound is very sensitive to hydrolysis. Adding excipients and coating the tablets possibly suppress this reaction.

Aspirin® was heated up to complete decomposition, leading to two main mass-loss steps (according to the TGA and DTG 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.

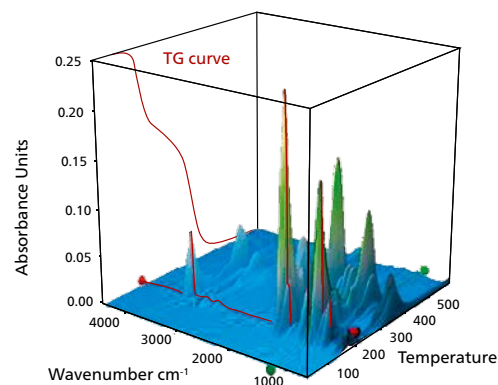
Stability, shelf life and residual solvents are essential in drugs and excipients



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.

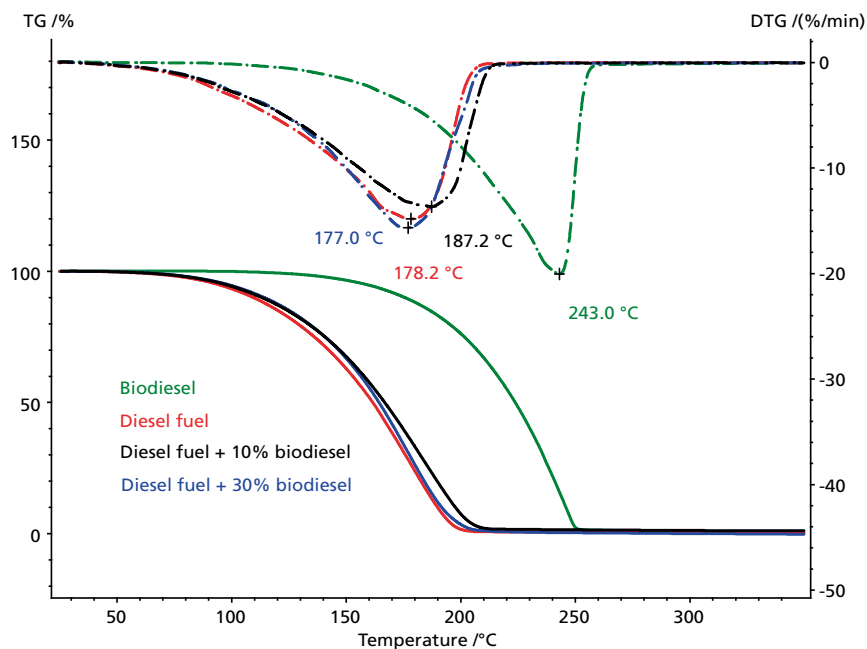


Measured FT-IR spectrum at 361 °C (red) compared to the database spectrum of salicylic acid (green).



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

## Differentiation between Diesel Fuel and Biodiesel

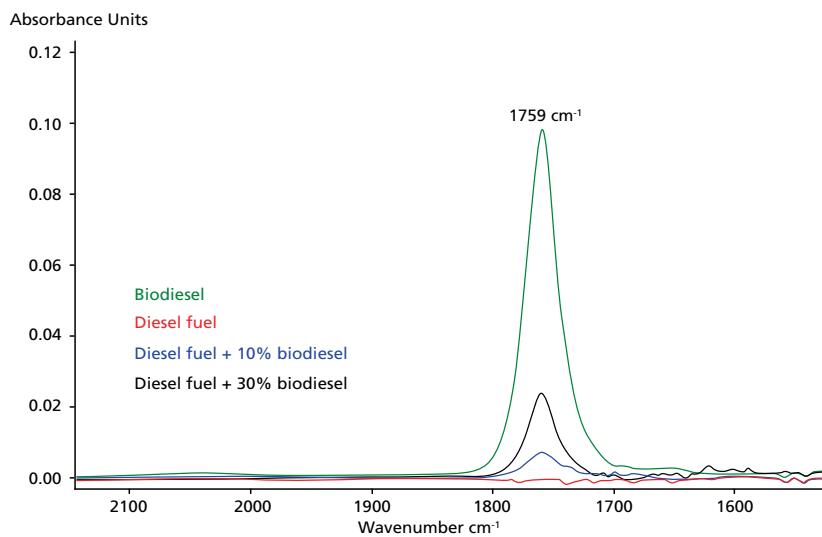


TGA comparison of various diesel fuel/biodiesel mixtures; sample masses: 10 to 11.5 mg, Al<sub>2</sub>O<sub>3</sub> crucibles, heating rate: 10 K/min, N<sub>2</sub> atmosphere

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<sup>-1</sup> 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.



Comparison of the IR spectra of all measured samples. Zoom in the region between 1500 and 2200 cm<sup>-1</sup>.



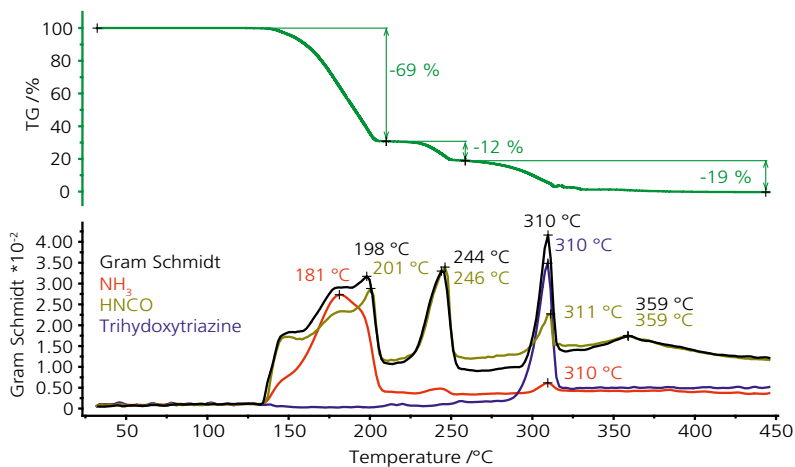
# Simulation of Application and

## Urea

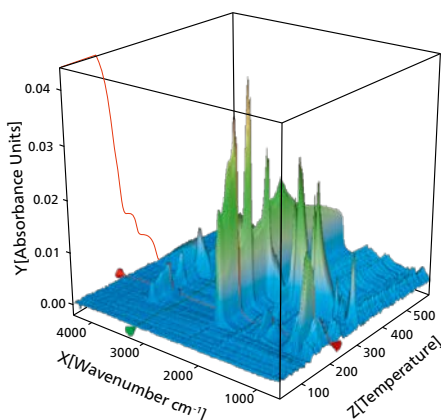
The use of urea can be diverse. It ranges from the production of melamine, to the use in fertilizers, to cosmetic and pharmaceutical applications. The information about thermal stability therefore plays an important role.

Investigations by TGA-FT-IR showed a three-step thermal decomposition. During the first mass-loss step ammonia and fulmic acid (HCNO) are formed, which could be detected by FT-IR. Above 200°C, fulmic acid reacts to cyanuric acid (trihydroxytriazine) which decomposes again to fulmic acid above 230°C. During the third mass-loss step, the complete cyanuric acid was detected in the gas phase. This is in good agreement with literature data.

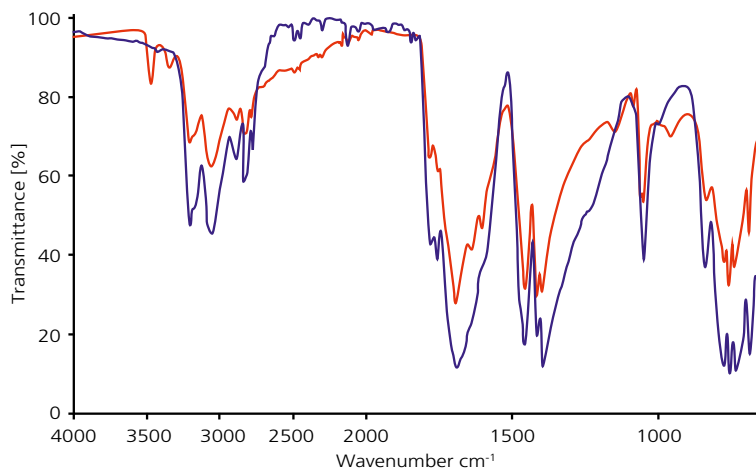
Cyanuric acid can also be detected by ATR-IR in the crucible if the reaction is stopped at 200°C.



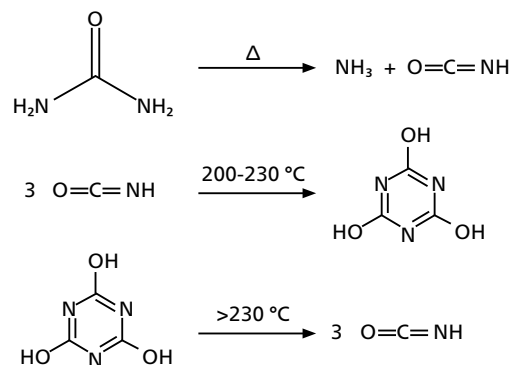
Measurement parameter: PERSEUS® TG 209 F1, RT – 450°C, nitrogen atmosphere, heating rate 5 K/min, gas flow 40 ml/min



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



ATR spectrum of crucible residue at 200°C (red) compared to the database spectrum of Trihydroxytriazine (blue)



# Manufacturing Conditions

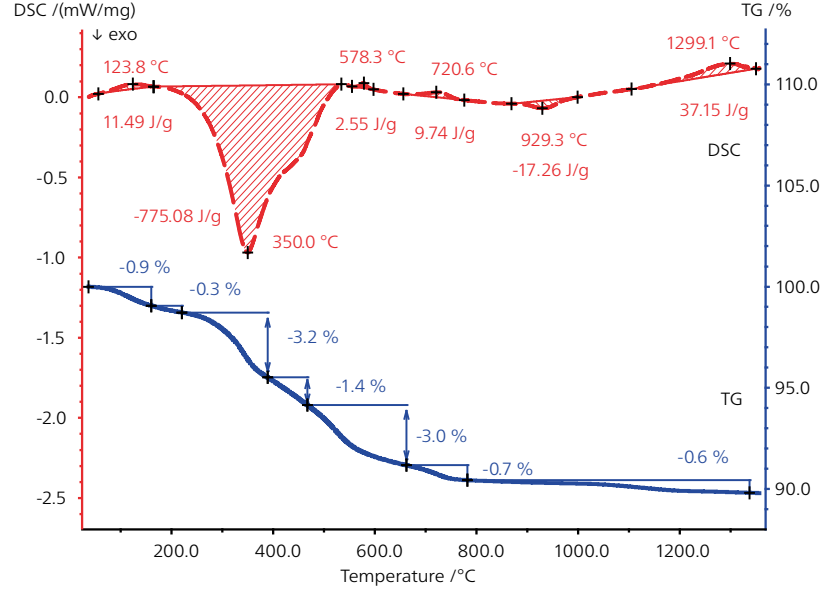
## Firing of Clay

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.

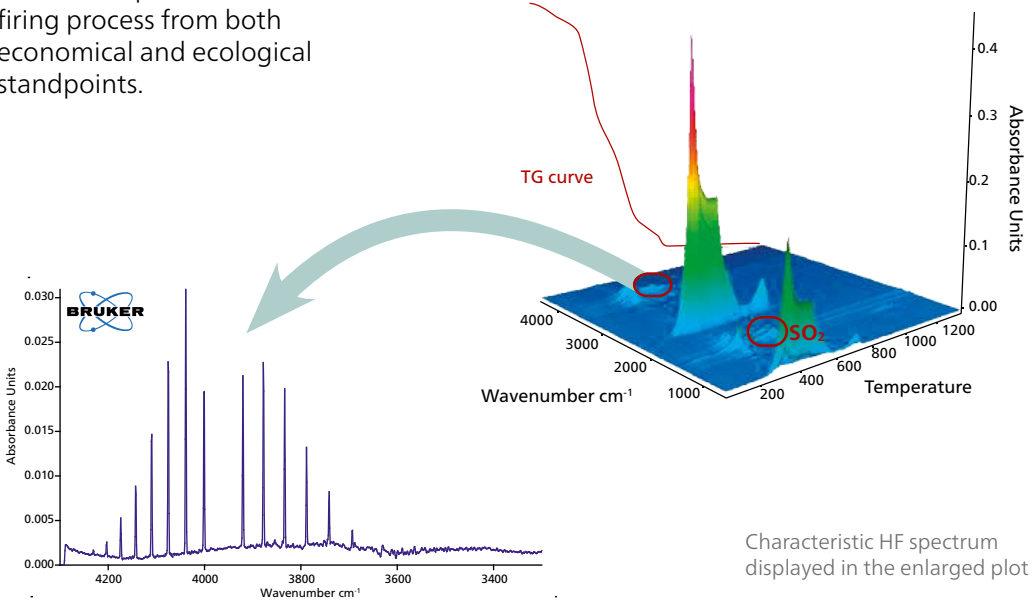
In this example, 107.6 mg of a clay green body was heated at a rate of 10 K/min in Pt/Rh crucibles in flowing air (50 ml/min). The predominant effect during heating is the burnout of the organics between 200°C and 550°C, which is accompanied by a high energy release (775 J/g).

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<sup>-1</sup>) and SO<sub>2</sub> (around 1300 to 1400 cm<sup>-1</sup>) 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)

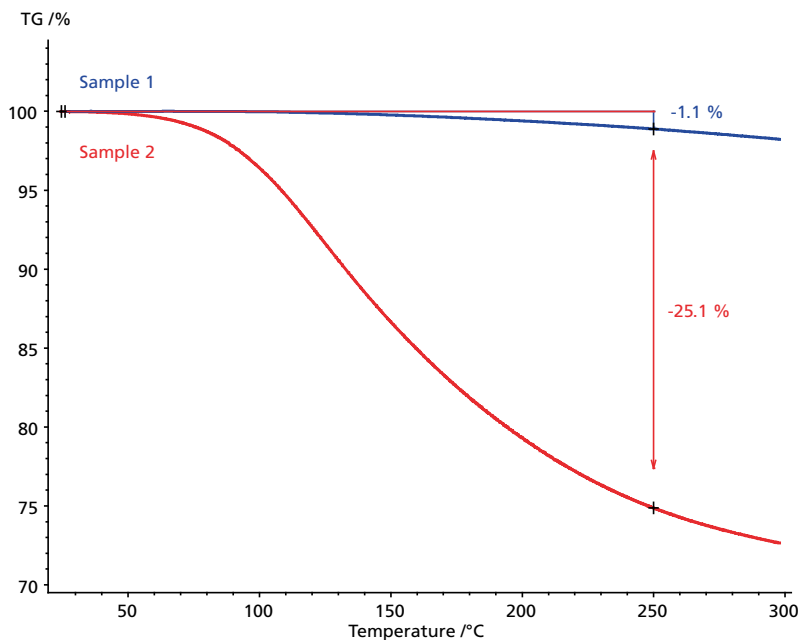


Characteristic HF spectrum displayed in the enlarged plot



TGA-FT-IR coupling can be of great help in detecting causes of failure during polymer processing.

# Process Optimization

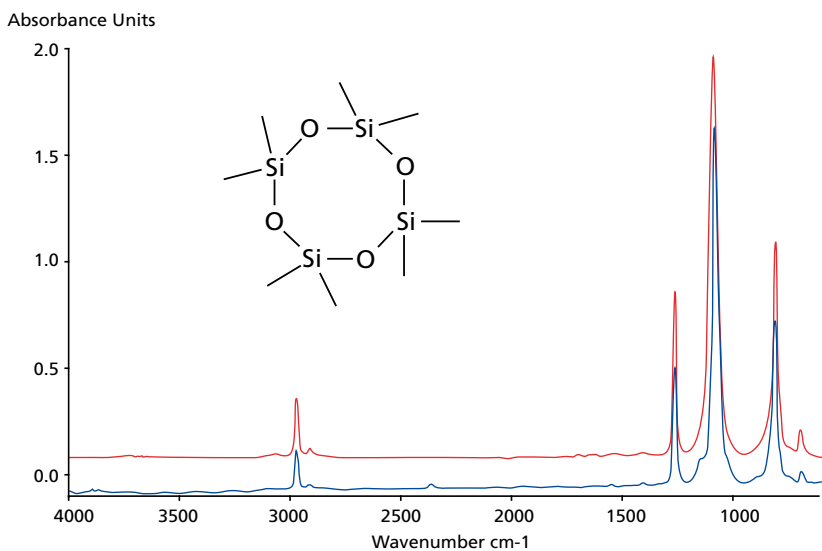


Comparison of the TGA results of two TGA-FT-IR experiments; sample masses: 24.5 mg and 26.3 mg, Al<sub>2</sub>O<sub>3</sub> crucibles, heating rate: 10 K/min, N<sub>2</sub> atmosphere

## Manufacturing of Silicones

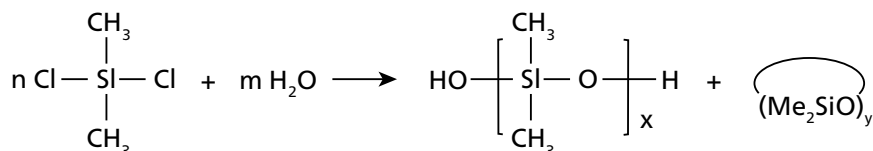
Here, two control samples of silicones were tested – one of them (sample 2) revealed manufacturing problems. The TGA curves exhibit significantly different thermal behavior. Sample 2 loses approx. 25% of its weight whereas the mass loss for sample 1 is only about 1% up to 250°C.

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



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

The polymerization mechanism for the generation of silicones often proceeds via cyclosiloxanes as intermediate products. Organochlorosilanes such as dimethylchlorosilane (CH<sub>3</sub>)<sub>2</sub>SiCl<sub>2</sub> are hydrolyzed and form silanoles which react at higher temperatures and in the presence of catalysts to become the desired 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.



Polymerisation mechanism of silicones

# Technical Specifications

## Characteristic Data for All Available FT-IR Coupling Systems


Coupling system	TG 209 <b>F1</b> ; TG 209 <b>F3</b>	DSC 404 <b>F1/F3</b> ; STA 2500 <i>Regulus</i> ; STA 449 <b>F1/F3/F5</b>	PERSEUS® STA 449; PERSEUS® TG 209 <b>F1</b>	TMA 402 <b>F1/F3</b> ; DIL 402 <i>Expedis</i> <i>Select/Supreme</i>	DSC 300 <i>Caliris</i> ®
Temperature range <sup>1</sup>	RT-1000°C ( <b>F3</b> ) 10°C (RT)-1100°C ( <b>F1</b> )	RT-1600°C ( <i>Regulus</i> , <b>F5</b> ) -150°C-2000°C ( <b>F1/F3</b> )	RT-2000°C (STA) RT-1100°C (TGA)	RT-1550°C (TMA) RT-2000°C (DIL)	RT-700°C
Additional automatic sample changer operation <sup>2</sup>	Yes ( <b>F1</b> )	Yes (DSC 404, STA 449) No ( <i>Regulus</i> )	Yes	No	Yes
Possible Bruker FT-IR spectrometer types <sup>3</sup>	INVENIO	INVENIO	ALPHA	INVENIO	INVENIO
Stand-alone operation of the FT-IR	Yes	Yes	Yes	Yes	Yes
Additional analytical instruments to be coupled	MS or GC-MS ( <b>F1</b> )	MS or GC-MS	MS or GC-MS	MS or GC-MS	MS or GC-MS
T <sub>max</sub> transfer line, transfer tube/ adapter head	400°C	230°C/300°C	250°C	230°C/300°C	400°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	11.8 ml	11.8 ml	5.8 ml	11.8 ml	11.8 ml
Detector <sup>4</sup>	DLaTGS or MCT	DLaTGS or MCT	DLaTGS	DLaTGS or MCT	DLaTGS or MCT

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

2 Larger Dewar recommended for MCT detector

3 For coupling with FT-IR spectrometers from other suppliers, please contact your NETZSCH representative

4 MCT detector requires LN<sub>2</sub> cooling



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