Thermal Analysis – Mass Spectrometer Coupling

Evolved Gas Analysis
Method, Techniques and Applications

Analyzing & Testing

Thermal Analysis and Evolved Gas Analysis

Thermoanalytical Techniques

Thermoanalytical techniques are universal tools for characterizing solids and liquids with respect to their thermal behavior. Especially Thermogravimetry and Simultaneous Thermal Analysis (STA, TGA-DTA/DSC) find broad application in testing the weight changes of a sample during a programmed heat treatment. This yields an abundance of information on material properties, composition and stability.

However, what is often lacking is chemical and analytical information about the course of events causing the mass change to the sample. Evolved Gas Analysis (EGA) by such techniques as quadrupole mass spectrometry can supply this additional information.

QMS 505 *Aëolos*

Application Fields for Thermal Analysis Coupled to Mass Spectrometry

Decomposition
- Water release
- Stability
- Residual solvent
- Pyrolysis

Solid-Gas Reactions
- Combustion
- Oxidation
- Adsorption
- Desorption
- Catalysis

Compositional Analysis
- Polymer content
- Proximate analysis
- Binder burnout
- Dewaxing
- Ash content

Identification
- Gas composition
- Fingerprint
- Partial pressure
- Fragmentation
- Solid-gas interactions

Evaporation
- Vapor pressure
- Sublimation

Reasons to Couple a Mass Spectrometer to a Thermal Analyzer

Complementary Information

Gas analysis allows for an explanation of the effects recorded by means of thermal analysis. The gases evolved can be detected down to the ppb range, which exceeds the typical sensitivity of thermoanalytical methods. The combination of the two methods therefore forms the ideal basis for top-notch material characterization.

Quadrupole Mass Spectrometry (QMS)

A quadrupole mass spectrometer's sensitivity, selectivity, speed and capacity for continuous operation make the system ideally suited for evolved gas analysis in combination with thermal analyzers, specifically Thermogravimetry (TGA) and Simultaneous Thermal Analysis (STA, TGA-DTA/DSC).

The efficient and reproducible ionization of gases in the electron impact ion source allows for high sensitivity in the detection of molecules, atoms and fragments.

HYPHENATION OF THERMAL ANALYSIS AND EVOLVED GAS ANALYSIS

NETZSCH offers complete solutions for Thermal Analysis coupled to Mass Spectrometry in terms of both hardware and software. Evaluation and presentation of the results are carried out with the well-proven *Proteus*® software. Gas flow conditions in all thermal analyzers are ideal for coupling to a mass spectrometer.

STA 509 *Jupiter*® coupled to QMS 505 *Aëolos*;
other thermal analyzers can also be coupled to MS, such as the TG 309 *Libra*®

The capillary system is designed for universal application. For the special application field of materials with high condensation tendency, such as metals, salts and high-boiling organics, NETZSCH offers a completely integrated coupling solution: The STA 449 ***F3*** *Jupiter*® with *SKIMMER* furnace.

This combination provides direct and simultaneous coupling of a thermal analyzer with a mass spectrometer, thus enabling gas transfer temperatures of up to 1950°C.

Ask your sales representative about upgrading your STA 449 ***F3*** *Jupiter*®.

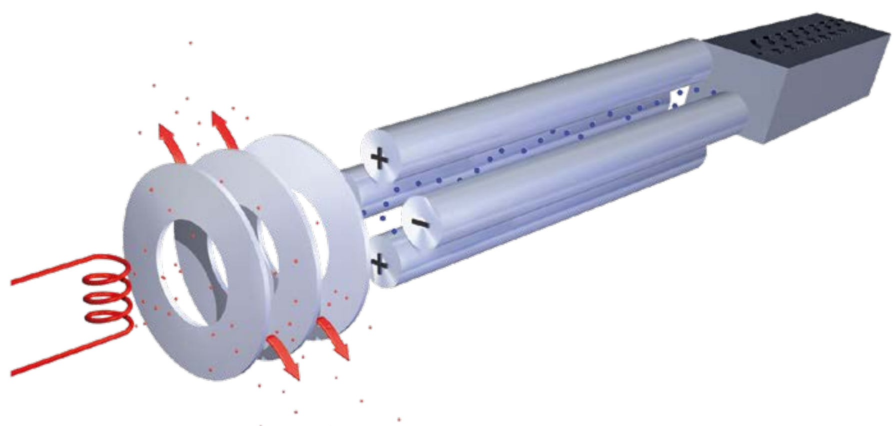
Withstanding the Test of Time – Capillary and *SKIMMER* Coupling Techniques

STA 449 ***F3*** *Jupiter*® with MS-*SKIMMER* system and a second furnace

TA-QMS Coupling Techniques

Interface for Pressure Adjustment

Mass spectrometers, composed of a mass filter, an electron impact ion source and an ion detector, work only in high vacuum. Therefore, an interface is required for the coupling of a thermobalance – which works with a purge gas flow at atmospheric pressure – to the mass spectrometer. Different versions of pressure reduction interfaces are realized depending on instrumentation and applications.



QMS 505 *Aëolos*

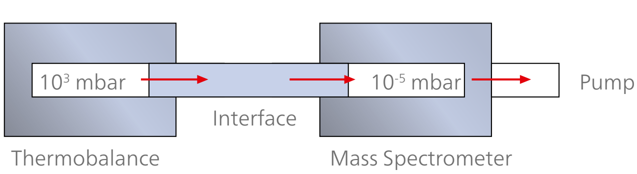
Single-Step Pressure Reduction

A capillary of small internal diameter connects the gas outlet on the furnace of the thermobalance with the gas inlet on the mass spectrometer. The pressure drops from atmospheric pressure down to high vacuum in one continuous step.

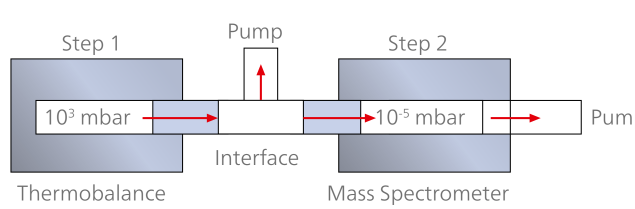
STA 449 ***F3*** *Jupiter*® with *SKIMMER* furnace

Double-Step Pressure Reduction

In this unique design, the first pressure-reduction step is located in the furnace, only a few millimeters above the sample. The second pressure-reduction step is located in the *SKIMMER* in the area of the compression zone formed behind the orifice.
In the *SKIMMER* furnace, an orifice system is used for the first pressure-reduction step for the ranges from 10^{-1} mbar to 10 mbar. As a second step, a *SKIMMER* is used as a molecular leak for the gas inlet into the high-vacuum recipient of the mass spectrometer.



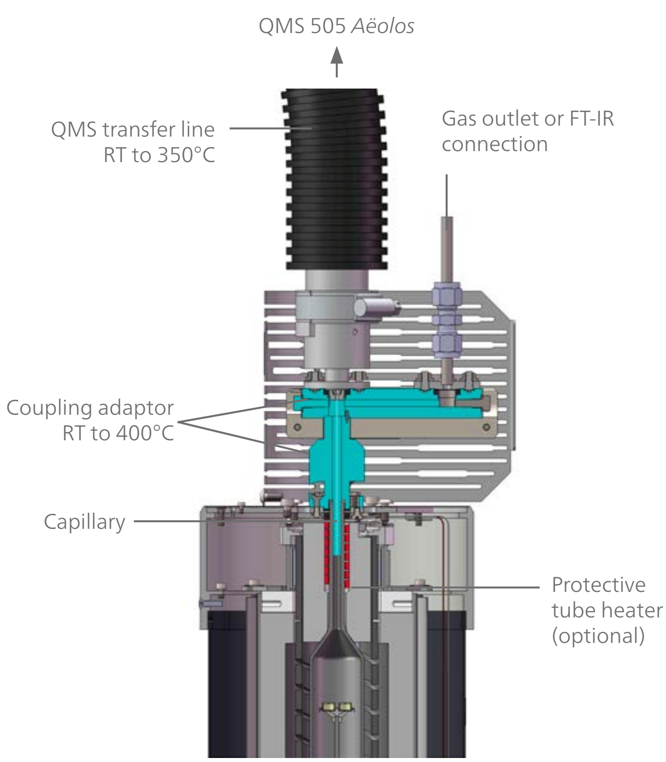
Capillary Coupling



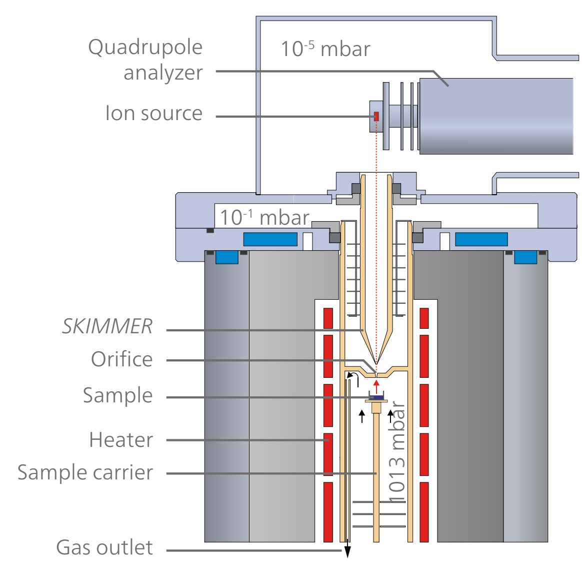
Double orifice *SKIMMER* coupling

Ideal Gas Flow Conditions Ensure Transport of All Relevant Gases

The aim of coupling is to have all relevant gases transported from the sample area into the ion source of the mass spectrometer for precise qualitative and quantitative analysis. This is only achieved through perfect gas flow conditions in the thermal analyzer, the coupling interface and the gas inlet of the mass spectrometer. As only a small quantity of gas is required for the analysis, a bypass is used at the gas outlet on the thermobalance for the excess purge gas flow; i.e., for the flow not passing through the coupling interface, which can be used for a second gas analyzer such as FT-IR.



Capillary coupling



Double orifice *SKIMMER* coupling

PERFECTLY COUPLED FOR PRECISE RESULTS.

TA-QMS 505 *Aëolos* Coupling

State-of-the-Art Capillary Coupling – Heating Throughout and Single-Step Pressure Reduction

The QMS 505 *Aëolos* quadrupole mass spectrometer features a fleshed-out design for capillary coupling to NETZSCH thermal analyzers (e.g., simultaneous TGA or STA). Volatile sample materials under controlled temperature treatment are directly transferred into the electron impact ion source of the MS via a fused silica capillary (optionally capillary made of stainless steel).

Optimized Capillary Coupling for Maximum Flexibility

- Minimization of cold spots in the transfer path
- Low probability of condensation due to an even temperature of 300°C (optionally 350°C) throughout the entire gas transfer system from the furnace outlet to the capillary to the MS gas inlet
- Flexible combination with standard thermoanalytical measurement methods (TGA, DSC, STA, DIL, etc.) along with the possibility of simultaneous coupling, e.g., MS-FT-IR.
- Robust and, at the same time, sensitive system with detection limit in the ppb-range
- Allows TGA-MS measurements under humid atmospheres
- Allows for the upgrade of existing thermal analyzers

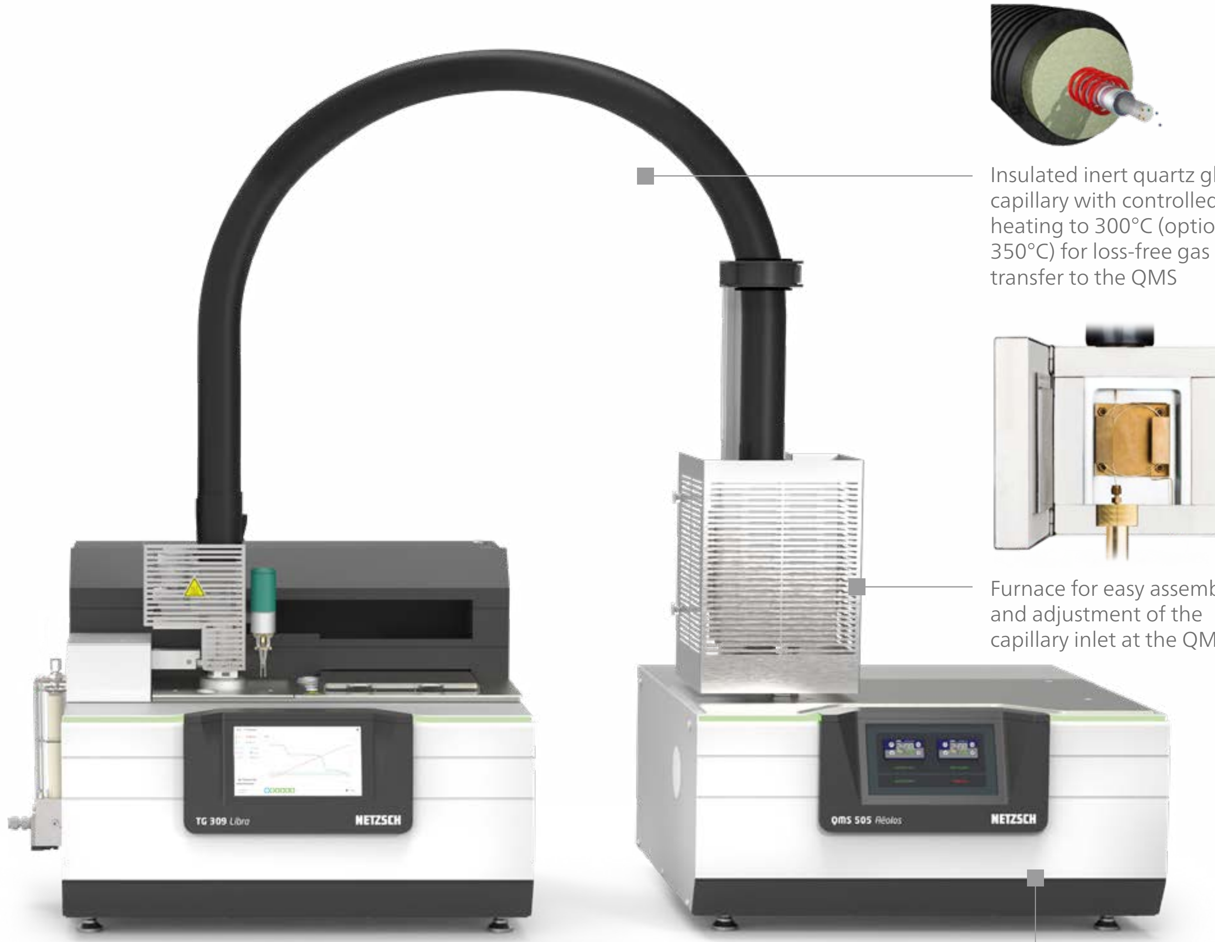
Hyperbolic Rod System

The hyperbolic rod system provides improved transmission and peak separation and corresponds exactly to the theoretical calculations (equations of motion) of the quadrupole.

- High transmission in high mass range
- Improved sensitivity in low mass range (H_2, He)
- Reduction of quadrupole contamination by optimized ion beam-guiding pre-filter

The QMS 505 *Aëolos* – the Perfect QMS for Gas Detection up to 300 u (Optionally 512 u)

- High peak stability over full mass range
- Hyperbolic quadrupole system with pre-filter
- SEM with discrete dynodes and integrated Faraday cup for high dynamic range and long lifetime
- EI source with two Y_2O_3-coated filaments
- 3-D presentation of MS and thermal analysis data
- Temperature of entire transfer system (incl. adapter) 300°C/350°C
- Internal reference for easy and fast calibration of the entire measuring system (mass axis, sensitivity, etc.) to different measurement conditions
- Operation and data evaluation with *Proteus®* software
- The *Aëolos* can also be independently employed for the analysis of other gas sources



Capillary Coupling Possibilities for the QMS 505 *Aëolos*

TGA-DSC/DTA Systems

- STA 509 *Jupiter® Supreme**: -150°C to 2000°C
- STA 509 *Jupiter® Select**: -150°C to 2000°C
- STA 509 *Jupiter® Classic*: RT to 1600°C

TGA Systems

- TG 309 *Libra® Select/Supreme*: RT to 1100°C

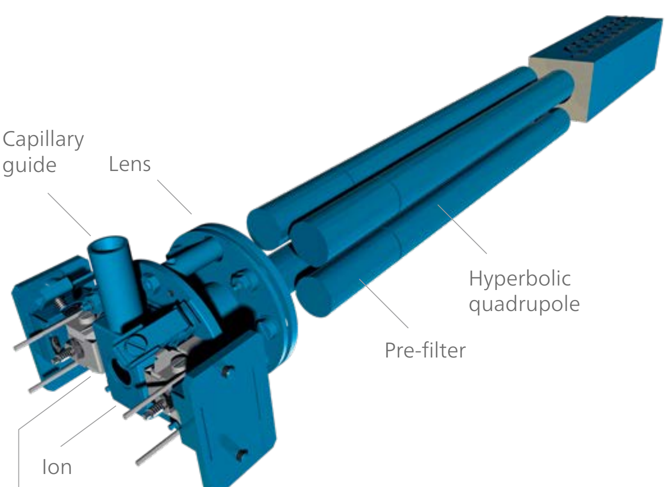
Dilatometer/Thermomechanical Analyzer

- DIL 402 *Expedis Supreme**: RT to 1600°C
- DIL 402 *Expedis Select*: RT to 1600°C
- TMA 402 ***F1/F3*** *Hyperion®*: -150°C to 1550°C

DSC Systems

- DSC 404 ***F1/F3*** *Pegasus®*: -150°C to 2000°C
- DSC 204 ***F1*** *Phoenix®*
- DSC 300 *Caliris® Supreme*

* The total temperature range depends on the furnace

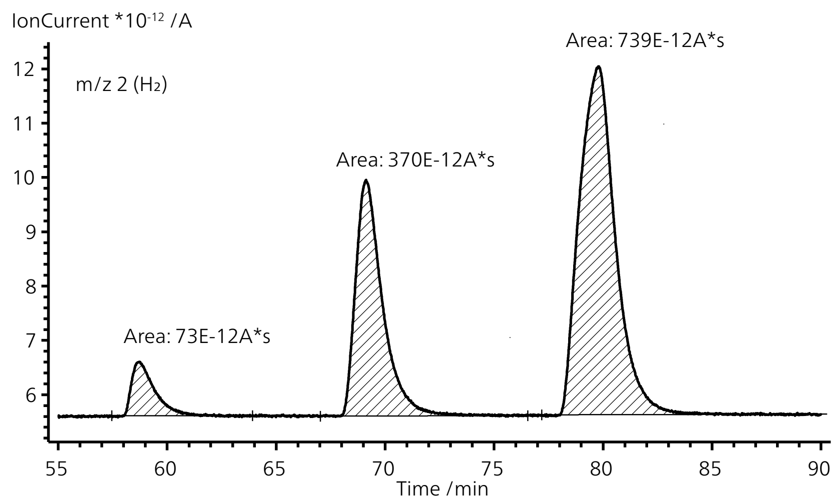


Coupling to existing NETZSCH devices of the series TGA, DSC, STA, DIL and TMA is possible. Please, contact your NETZSCH sales representative for details.

Sensitive and Linear Detector System

Detection Sensitivity for Hydrogen

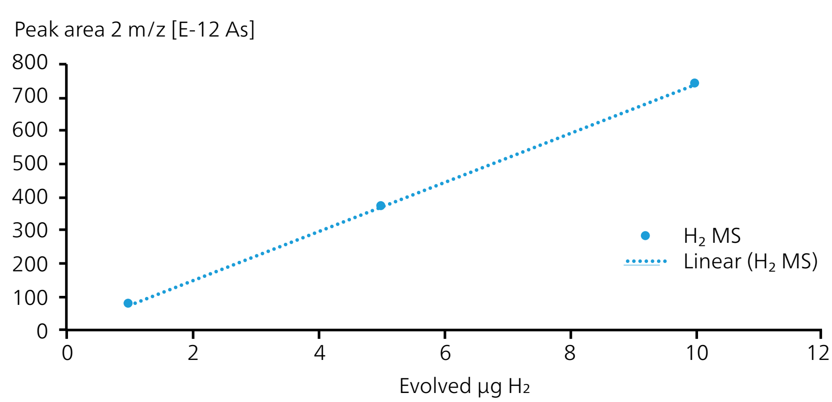
For demonstration of the detection sensitivity in the low mass range, argon purge gas was treated with pulses of hydrogen by using the NETZSCH *Pulse*TA®. The volume of the pulses was equivalent to 1 µg, 5 µg and 10 µg hydrogen. For m/z 2, an integration time of 1 s was used. Due to high performance of the MS, low hydrogen quantities can be detected with high precision.



STA-MS measurement of hydrogen pulses at constant temperature

Linearity of Hydrogen

Since the pulse area increases linearly with the pulse volume, quantification over a high concentration range is possible. The plot shown on the right, of the pulse areas in relation to the amount of gas, illustrates this linear correlation.



Presentation of the linear relationship between pulse area and the amount of hydrogen gas (H_2)

QMS 505 *Aëolos* Coupling

QMS Specifications

Mass range*	1 u to 300 u (optionally to 512 u); including auto-tuning using PFTBA to calibrate the mass scale axis
Mass filter	Quadrupole with hyperbolic rods and pre-filter (patented)
Ion source	Cross-beam EI ion source
Cathodes/filaments	Two iridium cathodes with Y_2O_3 coating
Electron energy	25 eV to 150 eV
Emission current	0.1 mA to 2 mA
Detector	SEM with discrete dynodes and integrated Faraday cup
Dynamic range (electronic)	9 decades
Detection limit	< 100 ppb (gas-dependent)
Resolution	0.5 u to 1.5 u
Vacuum system	Turbo molecular pump with 4-stage diaphragm pump (oil-free)
RF generator	High-stability fully digital RF generator
Measuring modes	Scan analog, scan bargraph, MID
Scan rate (electronic)	> 100 u/s (scan bargraph) → possible with reduced dynamics (10 u to 100 u, fixed measurement range of 1E-7, short settling and integration time; however, sufficient sensitivity for library search)
Power	115 - 230 VAC / 50 - 60 Hz
Power consumption	≈ 800 W

Transfer System from the Thermal Analyzer to MS

Adapter systems (STA/TGA/DSC/DIL to capillary and capillary to MS gas inlet system)	▪ Heated adapter and transfer line ▪ Temperature adjustable to T_{max} 400°C ▪ Single-step pressure reduction, no orifice
Entirely insulated capillary	▪ Made of quartz glass, max. 300°C, length ≈ 3 m, Ø 60 µm (Optionally made entirely of insulated stainless steel, max. 350°C, length » 2.5 m) ▪ Spare loop inside a furnace above the MS casing ▪ Can be changed out by the customer
Vacuum-tight connection between thermal analyzer and MS	Yes
Pressure reduction from thermal analyzer to MS	Single-step pressure reduction from 10^3 mbar to approx. $5x10^{-6}$ mbar

Technical Specifications

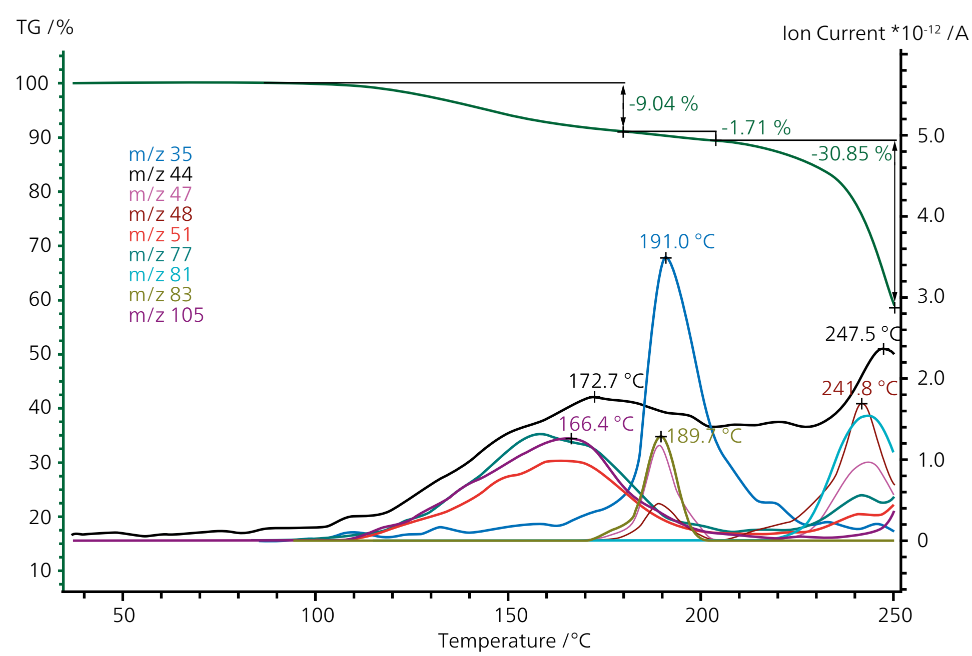
TA-QMS 505 *Aëolos*

Application

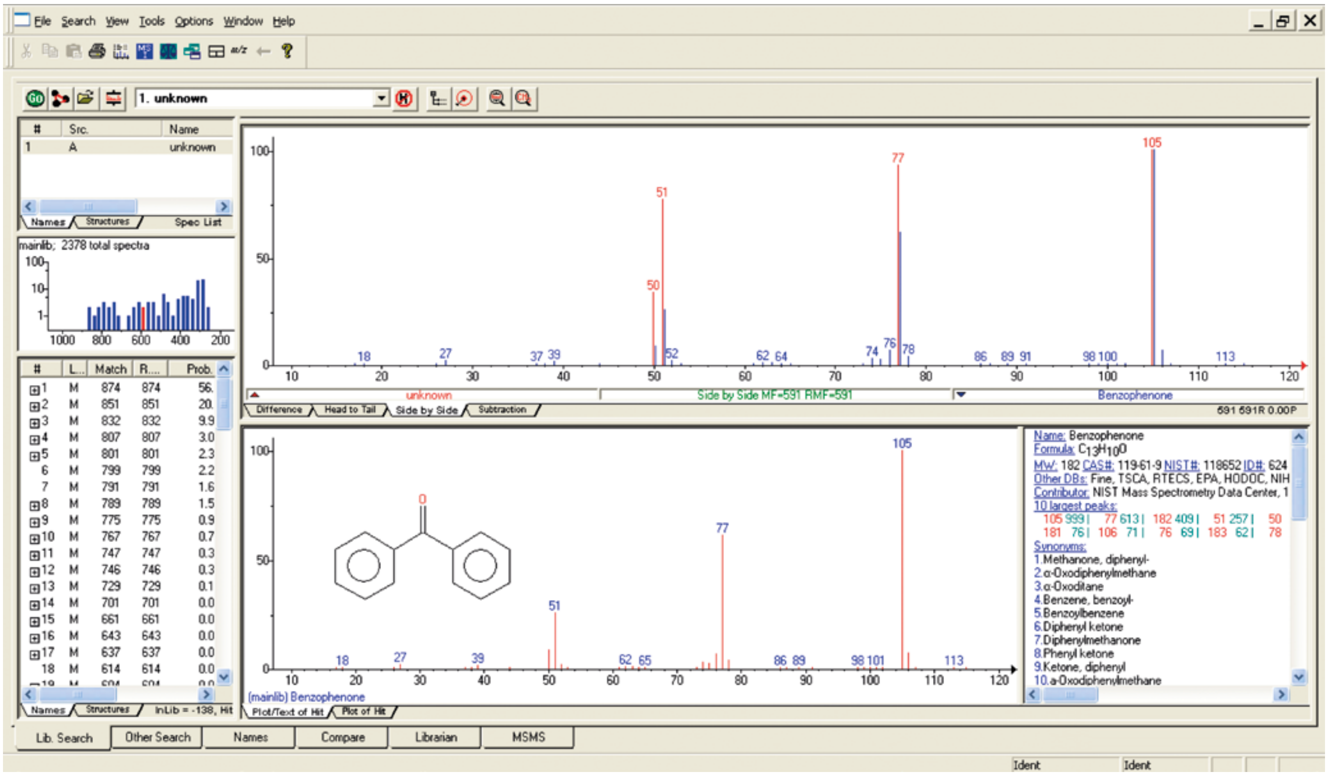
Measurement of an Unknown Polymer

This TGA-MS measurement of an unknown polymer (7.52 mg) was carried out in the temperature range between room temperature and 250°C in a helium atmosphere. The 3-D plot on the right below shows the TGA measurement together with the MS results. The plot below correlates the TGA curve with various MS traces of m/z 35, 47, 48, 51, 77, 83, and 105.

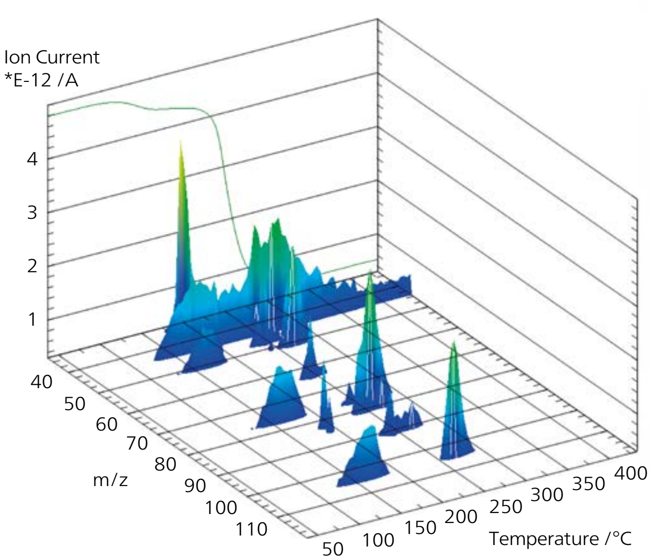
By exporting the 2-dimensional scan-bargraph directly into the NIST database (see figure on the right above as an example at 166°C), it becomes possible to interpret the individual mass-loss steps.



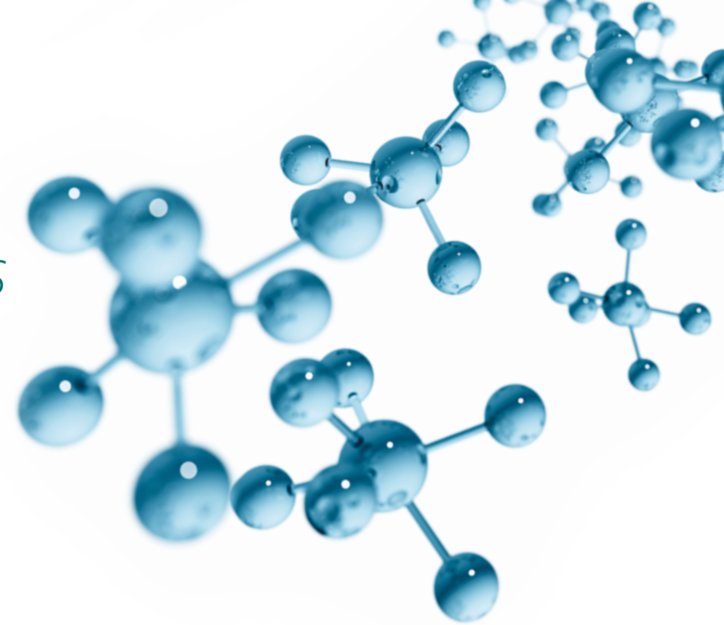
Correlation of the mass loss steps and the detected gases. m/z 35 can most likely assigned to HCl.



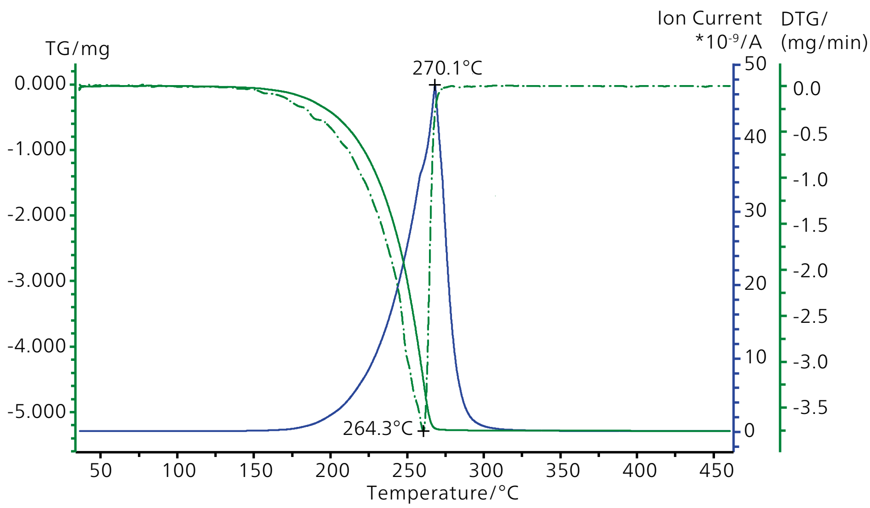
Direct export of the MS results at 166°C into the NIST database for identification of the evolved gases



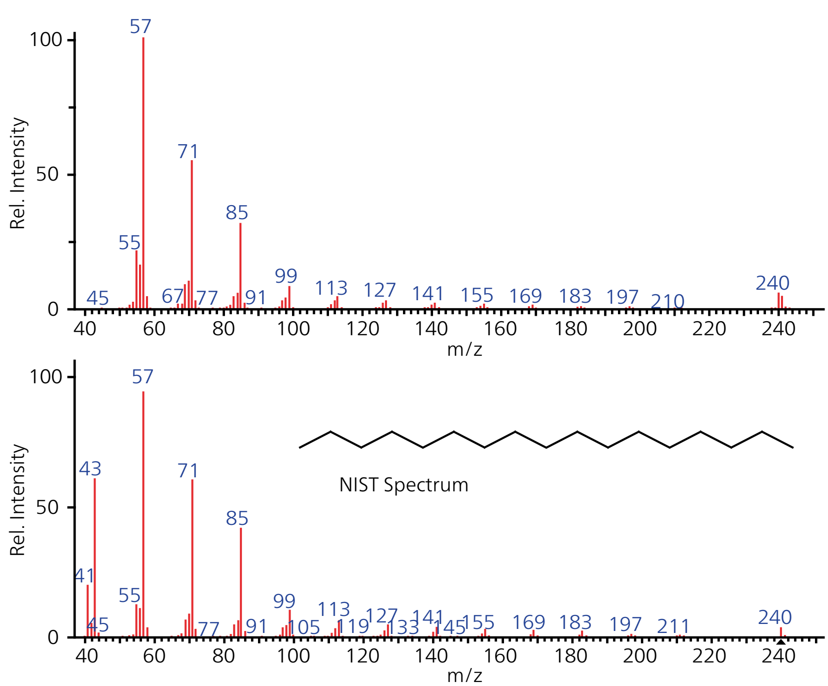
3-D plot of a TGA-MS measurement on an unknown polymer sample as a function of temperature



Continuous heating of the entire gas transfer line reduces the risk of condensation so that even larger molecules can be detected.



Temperature-dependent mass loss, mass loss rate and total ion current for evaporation of heptadecane



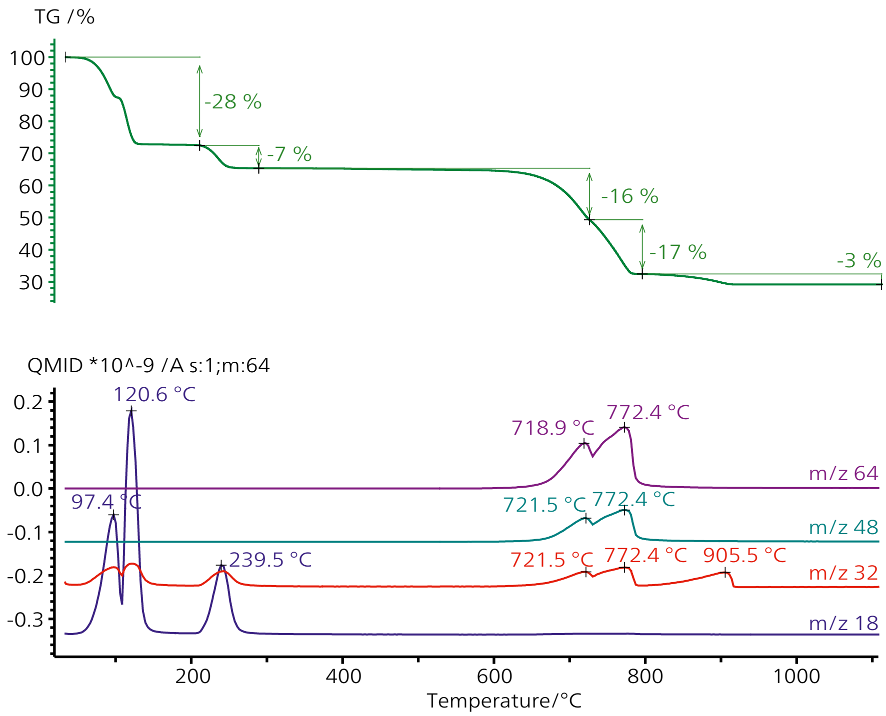
Comparison of the mass spectrum, measured at 270°C (top picture) with the spectrum of the NIST-MS library (picture below)

Analysis of high boiling organics – Heptadecane

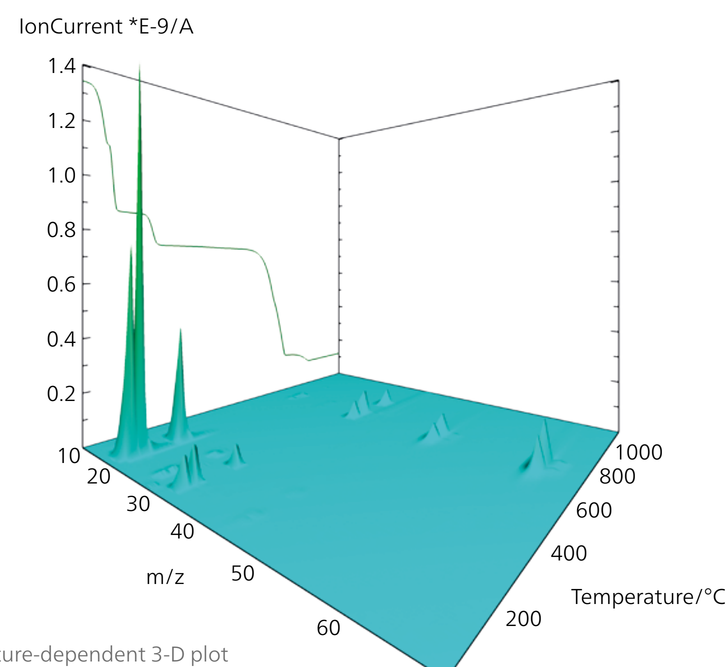
This measurement was carried out with the STA coupled to the QMS *Aëolos*. The temperature adapter, transfer line and MS inlet were set to 300°C.

Evaporation of heptadecane ($CH_3(CH_2)_{15}CH_3$; bp. 302°C) starts at approx. 170°C (blue curve). The maximum decomposition rate is achieved at 264.3°C (DTG peak, dotted line) when using a heating rate of 20 K/min. After a short delay, the ion current achieves its maximum at 270.1°C (dark blue curve). After detection of heptadecane in the MS, the ion current immediately returns to the zero-level without any significant tailing effect.

A comparison between the detected MS spectrum for heptadecane and the corresponding NIST library spectrum confirms that even larger molecules (e.g., m/z 240) pass the adapter, transfer line and MS inlet without condensation.



Thermogram of blue vitriol in combination with the ion current curves of m/z 18, 32, 48 and 64



Temperature-dependent 3-D plot of blue vitriol with TGA curve in the background

TGA-MS Measurement on Blue Vitriol ($CuSO_4$*5 H_2O)

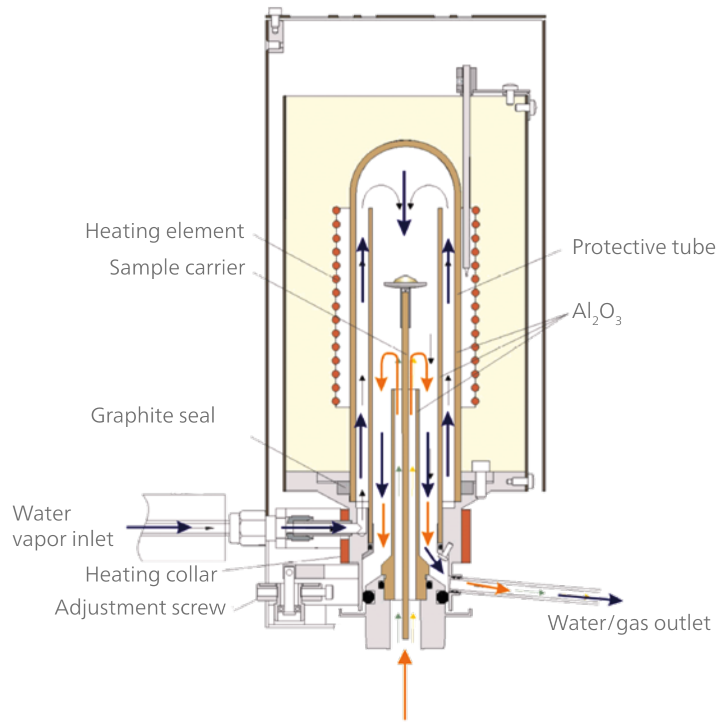
The mineral sample of blue vitriol (21.0 mg) shows a stepwise release of its crystalline water content (5 * H_2O) in the temperature range below 400°C. The peaks (97°C, 120°C, 240°C) in the ion current of m/z 18 illustrates these individual steps. In the higher temperature range, the water-free sample further decomposes to copper-(I)-oxide while releasing sulfur dioxide (m/z 32, 48, 64) and oxygen (m/z 32).

ACCESSORIES MAKE THE DIFFERENCE

Water-Vapor Furnace for High Water-Vapor Concentrations

STA-MS Measurements under Water-Vapor Atmosphere

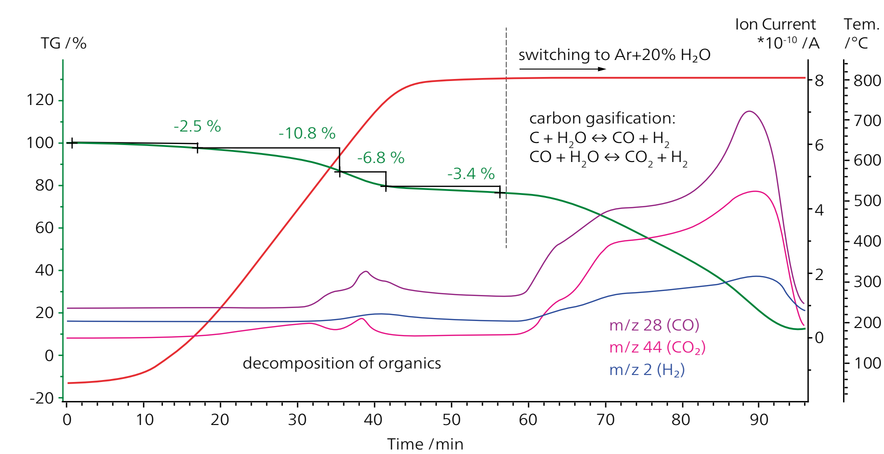
In addition to a variety of other furnaces for the STA systems, NETZSCH also offers a water-vapor furnace which allows for hydrous atmospheres all the way through pure vapor at the sample between room temperature and 1250°C. The water-vapor furnace can be connected to a humidity or a water-vapor generator. It provides protection against flooding and minimal dilution due to a special gas flow design.



Cross-section of the water vapor furnace

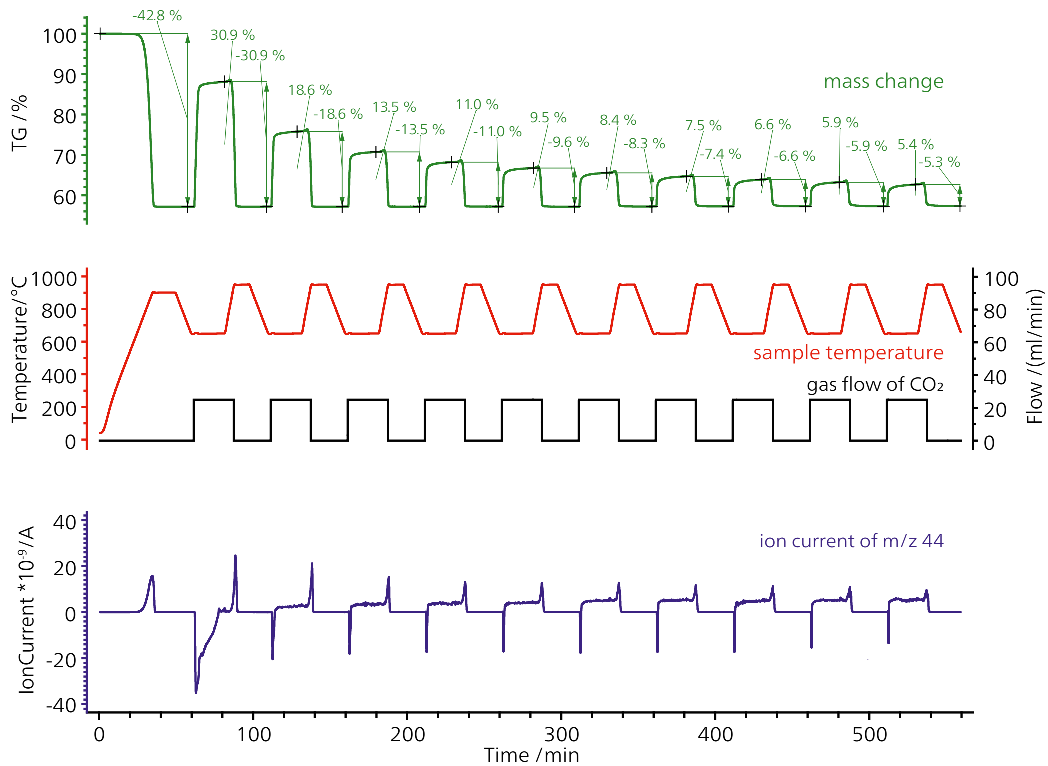
Monitoring Reaction Steps During the Carbon Gasification Process

This plot shows a typical example of the configuration with the water-vapor furnace: Here, the water vapor serves as a reactant for the transition from coal to hydrogen, and the STA with coupled gas analysis shows both the weight loss of the coal sample and the products resulting from the reaction.



Time-dependent presentation of the release of CO, CO_2 and H_2 during carbon gasification in hydrous atmosphere (TGA curve in green, temperature curve in red)

Investigation of Sorbent Activity of Limestone by Carbonation-Calcination Cycles

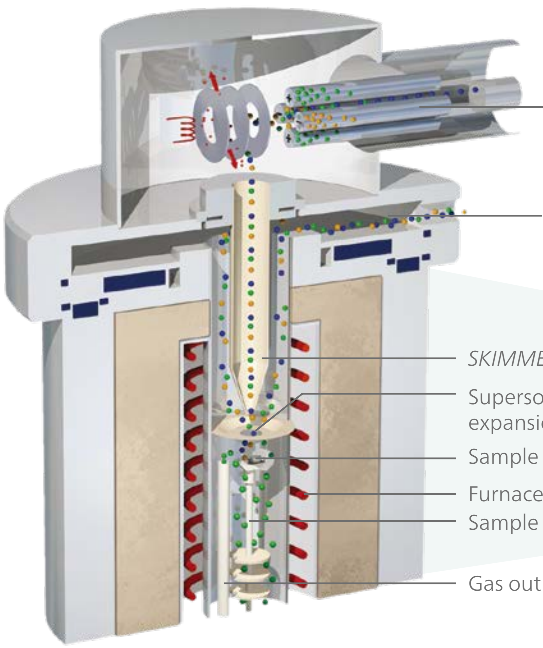


10 carbonization cycles on limestone

The inconsistent nature of some renewable energy forms, such as solar and wind, leads to an increased interest in Thermochemical Energy Storage (TES). The TGA is a suitable tool for investigating the cycle stability of these TES systems. In this example, a natural limestone sample (61.9 mg) was treated with 10 subsequent carbonation-calcination cycles. The observed mass changes within the TGA curve show the stabilization of the sorbent activity of the limestone, which takes place in the course of the individual cycles. In addition, MS analysis provides a further tool for evaluating the sample behavior as the consumption or release of CO_2 in each reaction step is monitored.

SKIMMER

DOUBLE ORIFICE SOLUTION FOR HIGH-BOILING MATERIALS



Shortest possible coupling solution via special supersonic jet gas transfer

Vertical – Top-Loading STA System and *SKIMMER* on Top of the Furnace

The *SKIMMER* coupling (realized with the STA 449 ***F3*** *Jupiter*®) actualizes the shortest possible route for gas transfer from the sample to the QMS. The *SKIMMER* collimates the molecules from the zylindric-shaped jet expansion behind the divergent nozzle towards the QMS ion source.

The pressure reduction from atmospheric pressure all the way up to high vacuum behind the *SKIMMER* orifice is achieved in two steps along a distance of less than 20 mm. This drastically reduces the risk of condensation and thus achieves high detection sensitivity. Even metal vapors are detected by this special coupling system.

The nozzle and *SKIMMER* are precisely machined from either alumina or glassy carbon, allowing application temperatures of 1450°C or 1950°C in the corresponding furnaces. The molecular beams are analyzed by a quadrupole mass spectrometer up to high mass numbers of 512 u.

It is possible to upgrade an existing STA 449 ***F3*** *Jupiter*® instrument. The upgrade process will be assisted by your local sales professional. Please contact us for further information.

STA 449 ***F3** Jupiter®* – *SKIMMER* Coupling

STA 449 ***F3** Jupiter®* Specifications	
Temperature range	RT to 2000°C
Furnaces and double hoist positions (left and right)	*SKIMMER* furnace in left position: ▪ SiC: RT to 1450°C with *SKIMMER* orifice made of alumina ▪ Graphite: RT to 1950°C with *SKIMMER* orifice made of glassy carbon Right position: For an exchangeable 2nd furnace (temperature range -150°C to 1650°C)
Weighing range	35 g
Sensor types	Quickly exchangeable sensors for different measurement methods: ▪ TGA ▪ TGA-DTA ▪ TGA-DSC
Gas flow measurement	3 mass flow controllers (MFC), optional 4 MFCs
Vacuum-tight	10^{-4} mbar (10^{-2} Pa)
Oxygen Trap System *OTS*™	Optional; O_2 partial pressure < 1ppm
Crucibles in various dimensions	Pt, Al_2O_3, graphite, Au, SiO_2, Cu, W, Ag, BN, ZrO_2, Pt with Al_2O_3 liner, Al incl. with lids pierced (50 µm hole), etc.; more on request

QMS Specifications	
Mass range	1 u ... 512 u
Mass filter	Quadrupole
Ion source	Electron impact, energy up to 125 eV, adjustable in steps of 1 eV for “soft” and “hard” ionization
Cathodes/filaments	Iridium cathodes with yttrium
Detector	Faraday; SEM
Operating pressure	< 10^{-5} mbar (Faraday); < $5x10^{-6}$ mbar (SEM)
Detection limit	<100 ppb (gas-dependent, measured with toluene)
Vacuum	$5x10^{-6}$ mbar
Measuring modes/ scan rates	▪ Analog scan: 10 ms/u …60 s/u ▪ Scan bargraph: 2 ms/u …60 s/u ▪ Multi Ion Detection (MID): 0.5 ms/u ... 60 s/u; up to 64 selectable mass numbers and mass ranges

SKIMMER Coupling Specifications	
Arrangement	▪ Vertical ▪ Completely heated
1st pressure reduction step	**Orifice**
Materials	▪ Polycrystalline alumina (1450°C) ▪ Glassy carbon (1950°C)
Vacuum system	Pump system and pressure control for constant sensitivity in MS
2nd pressure reduction step	***SKIMMER* cone**
Materials	▪ Polycrystalline alumina (1450°C) ▪ Glassy carbon (1950°C)

Technical Specifications

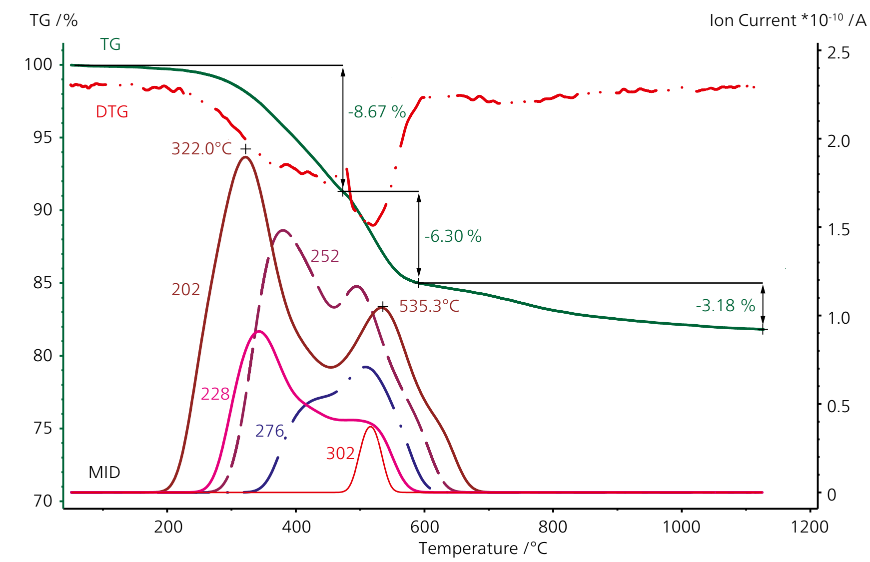
SKIMMER Applications

Sophisticated applications require ingenious analytical tools

Decomposition Products of Carbon Pitch

Carbon pitch is the primary product from the distillation of coal tar. It is used as a binding agent in the production of carbon anodes for aluminum smelters and graphite electrodes for electric arc furnace steel producers.

During thermal treatment of carbon pitch powder (55.2 mg) in a nitrogen flow (50 ml/min) below 600°C, mainly aromatic compounds of high molecular weight are released. A selection is shown here with MID curves for pyrene (m/z = 202), triphenylene (m/z = 228), benzo(a)pyrene (m/z = 252), benzo(ghi)perylene (m/z = 276) and dibenzopyrene (m/z = 302).

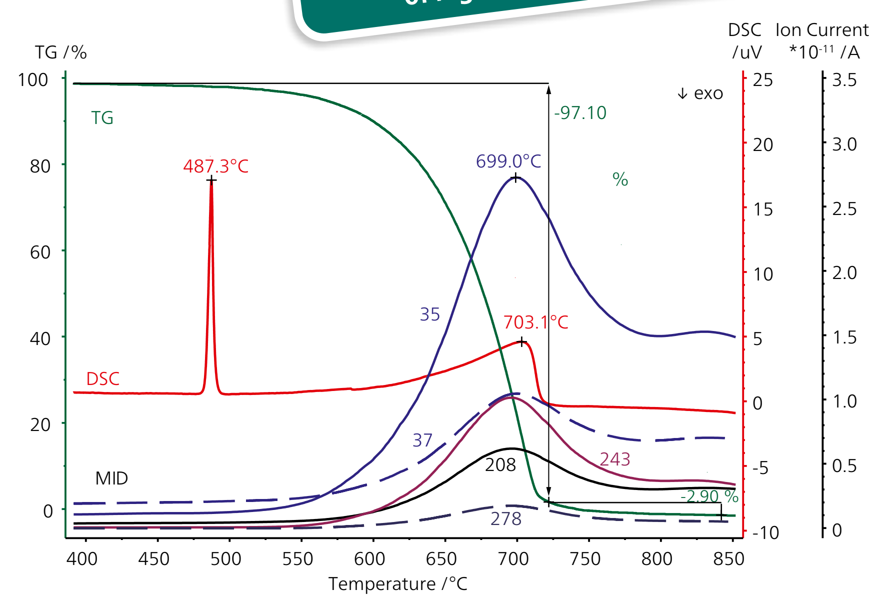


Comparison of the temperature-dependent course of mass loss and mass-loss rate of carbon pitch with ion current curves of typical mass numbers of aromatic compounds

Detection of aromatic compounds of high molecular weight

Detection of Salt Vapors

The measurement on lead-(II)-chloride (7.92 mg) in an argon flow of 150 ml/min exhibits evaporation starting in the melting range (487°C). The molecule ion ($PbCl_2$ m/z 278) and fragment ions caused by dissociation and ionization (PbCl m/z = 243, Pb m/z = 208, Cl m/z 37, Cl m/z = 35) are clearly detected far below the boiling temperature (950°C) of the starting material.

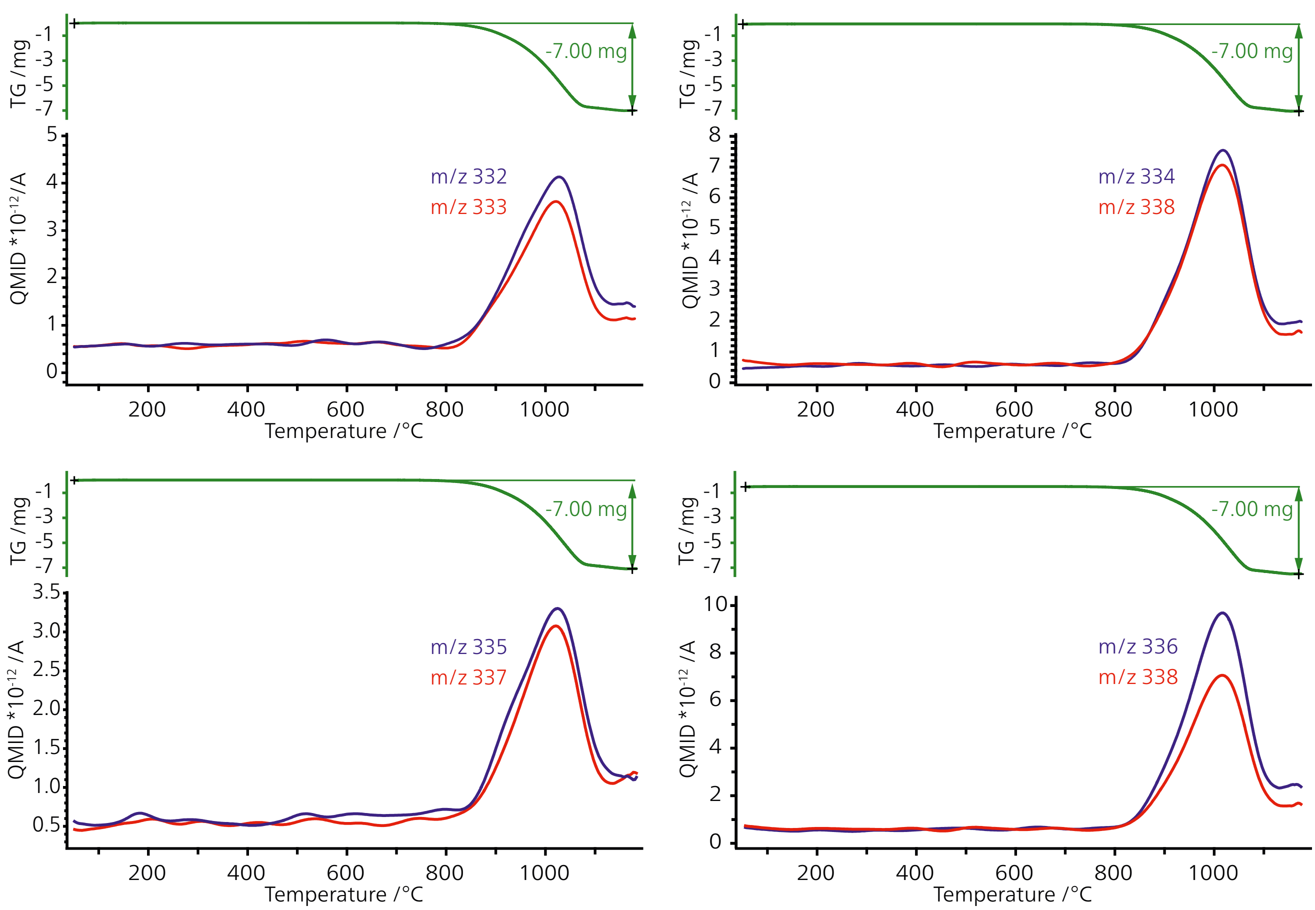


Comparison of the mass loss and DSC curve of $PbCl_2$ with the ion current curves of the fragment ions

Thermal Stability of the Thermoelectric Material PbTe

Knowledge about thermal stability properties, such as phase change and evolving gases at elevated temperatures, is crucial for the development of thermoelectric materials. In this example, the thermal stability of PbTe was analyzed using the STA 449 ***F3*** *Jupiter*® coupled to a mass spectrometer via the *SKIMMER* system.
The plots each show the TGA curve of the PbTe sample, but with different mass numbers. PbTe starts decomposing at around 600°C. The plotted mass numbers represent the combination of the Pb and Te isotopes. The following gaseous products were detected:

Fragment Mass (m/z)	Composition of the PbTe Isotopes	
332	$^{207}Pb + ^{125}Te$	$^{206}Pb + ^{126}Te$
333	$^{208}Pb + ^{125}Te$	$^{207}Pb + ^{126}Te$
334	$^{208}Pb + ^{126}Te$	$^{206}Pb + ^{128}Te$
335	$^{207}Pb + ^{128}Te$	
336	$^{208}Pb + ^{128}Te$	
337	$^{207}Pb + ^{130}Te$	
338	$^{208}Pb + ^{130}Te$	



Correlation of the ion current (QMID) of fragment masses m/z 332 to 338 with the mass-loss curve (TGA) of PbTe

Proteus®

The Software for Performing Simultaneous Measurements Using Thermal Analysis Coupled with the QMS 505 Aëolos

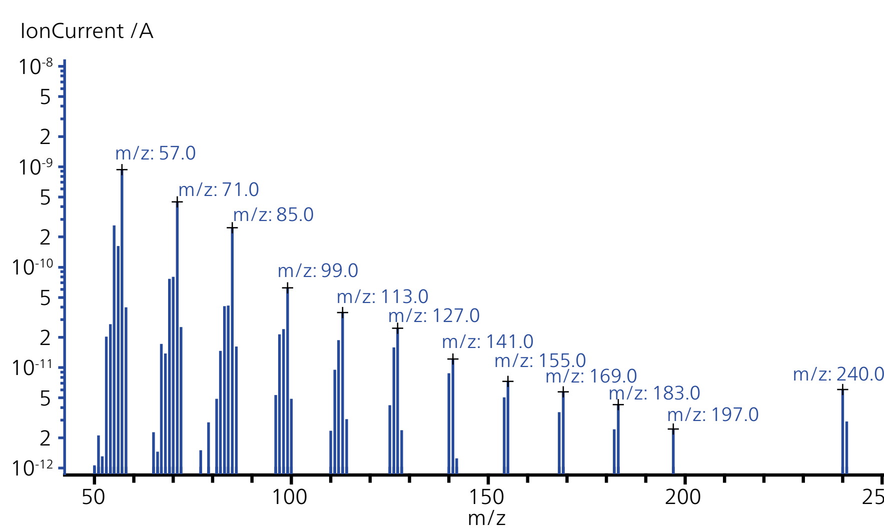
Measurements using STA/TGA/DSC/DIL instruments coupled to the QMS 505 *Aëolos* can be controlled entirely via *Proteus®* software*, which combines the measurement and analysis software of the two coupled methods into a single software application for both control and data acquisition. *Proteus®* allows for the individual definition of any parameters relevant to thermal analysis (e.g., temperature program, heating rate, etc.) as well as any parameters relevant to the mass spectrometer (e.g., mass ranges, scans, etc.). For hyphenated measurements, the two systems (STA/TGA/DSC/DIL and QMS 505 *Aëolos*) are started and stopped simultaneously.

During the measurement, the thermoanalytical and MS data are displayed in a common plot and stored in one measurement file. This is then used in the *Proteus®* analysis software for joint presentation and evaluation. There is no longer any need for complicated data import or switching between different applications.

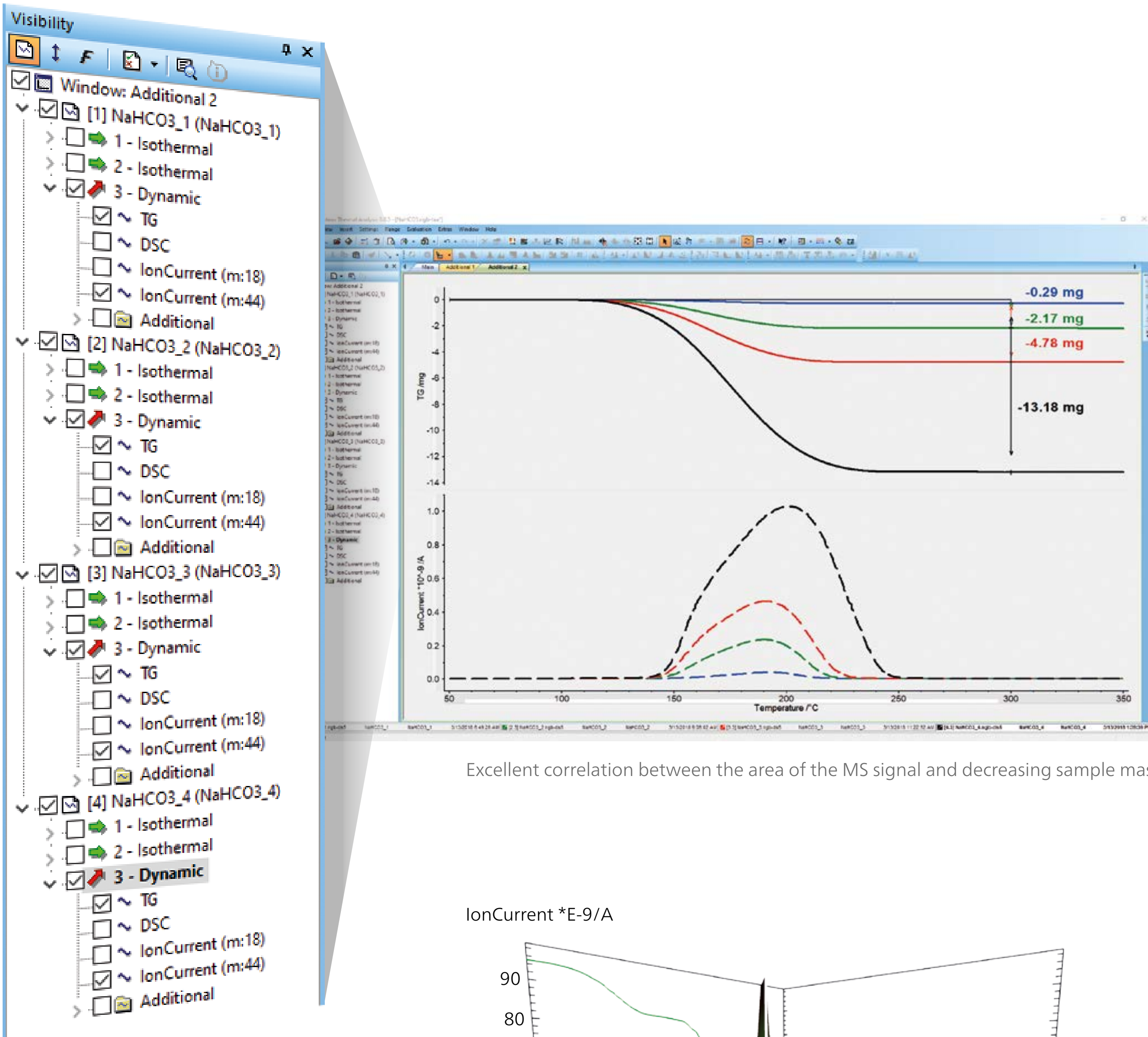
* Available for capillary mass spectrometer (QMS 505 *Aëolos*)

Comprehensive Information via Scan-Bargraph

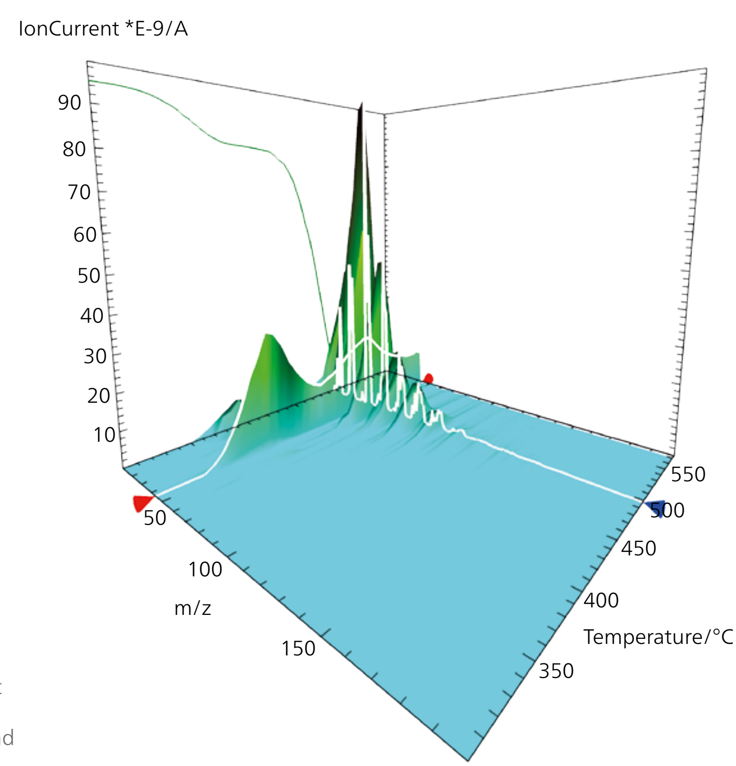
A scan-bargraph is often the basis for depicting comprehensive information about all of a sample's evolved species; it allows for displaying a selection of all mass numbers or just individual ones of interest in *Proteus®* software as continuous MID curves. Here, one of the repeated scans is shown for heptadecane measured in argon.



Significant fragmentation pattern of heptadecane at 250°C



Excellent correlation between the area of the MS signal and decreasing sample mass



The 3D plot of temperature, m/z ratio and ion current depicts all MS data within one graph. It enables visualization of the relationship between mass loss and the signal increase in the individual m/z numbers.

Key Software Features

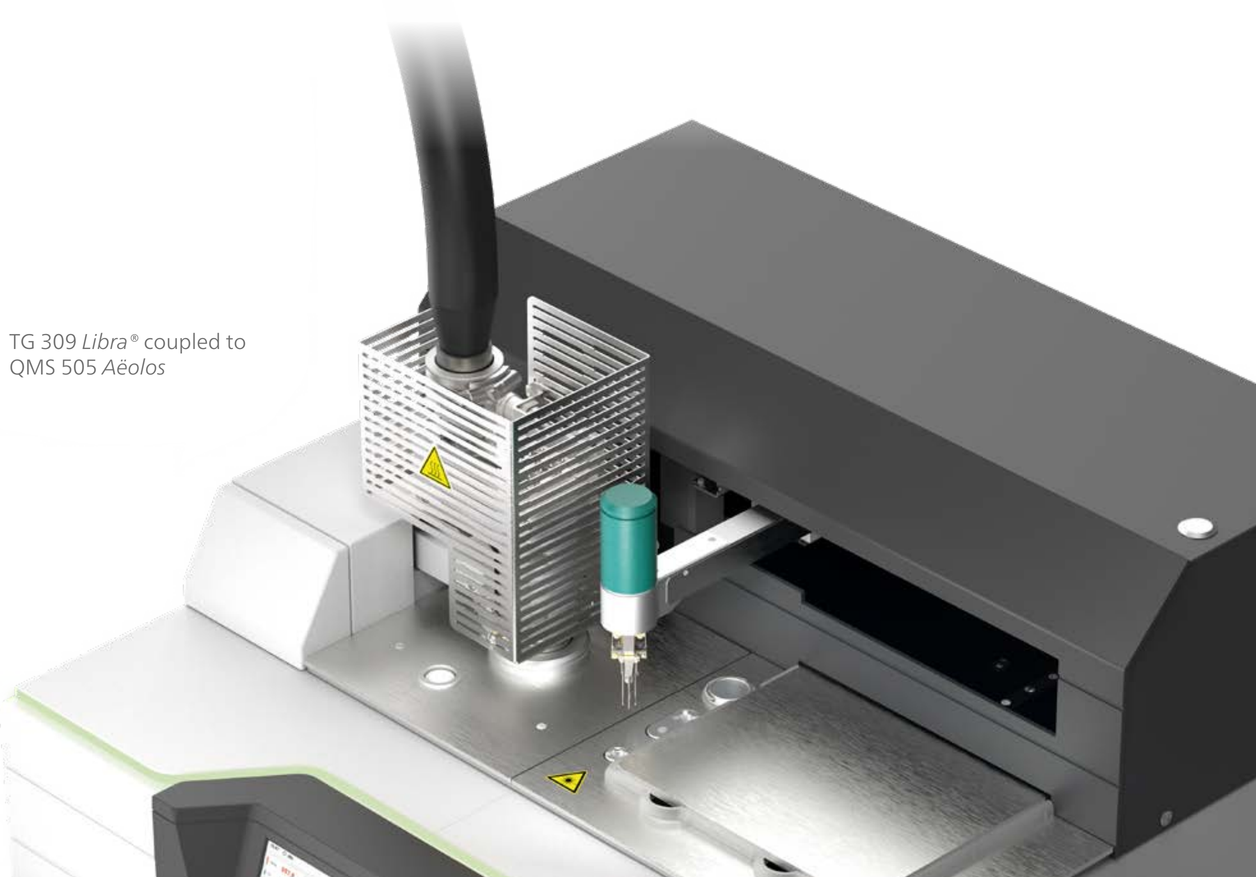
Measurement

- Complete integration of thermal analysis and QMS software into *Proteus*®*
- Method-based measurement and evaluation*
- Simultaneous start/stop of the coupled measurements
- Three different scan modes: scan analog, scan bargraph, MID
- Selection of different scan bargraph ranges at the same time
- Selection of scan bargraph or scan analog with optimized rate and sensitivity in different channels
- Individual MS parameters for each position of the automatic sample changer*

Analysis

- Evaluation of MS results within *Proteus*®
- Evaluation of results precisely correlated in terms of time and temperature
- Presentation of MS signals (TIC and individual mass numbers) together with thermoanalytical curve
- 3-D presentation of spectra data together with temperature, TGA and/or DSC curves and single mass number traces, including peak determination, different color schemes, and surface views
- Easy extraction of 2-D MS data from 3-D plot for database comparison
- Spectra export in NIST format for identification in the NIST database

* Available for capillary mass spectrometer (QMS 505 *Aëolos*)



TG 309 *Libra*® coupled to QMS 505 *Aëolos*

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, both before and after sale. Our qualified personnel from the technical service and application departments are always available for consultation. In special training programs tailored for you and your employees, you will learn to tap the full potential of your instrument. Choose your preferred training method: Online, on-site or at our NETZSCH training center.

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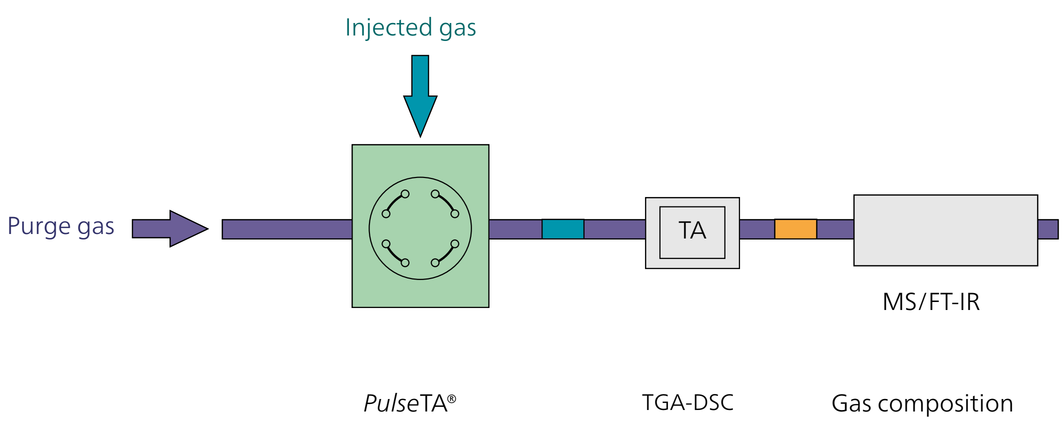
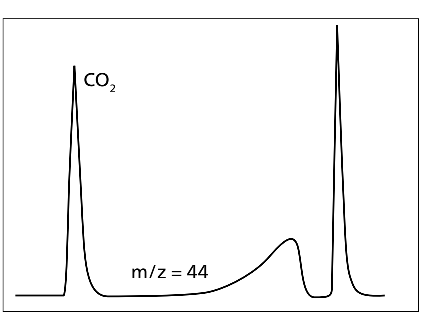
*Pulse*TA®

Calibration/Quantification – An Accessory for MS Capillary Coupling

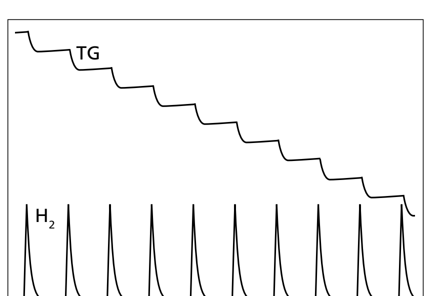
The quantification of MS signals requires calibration of the whole coupled system with a known type and amount of gas or solvent to control for the temperature-dependent flow properties. *Pulse*TA® is a perfect tool for achieving quantitative gas detection in separate calibration runs or even online during a sample measurement. A known amount of gas is injected into the sample gas stream and the registered signal of the resulting pulse is integrated. The application of *Pulse*TA® also allows for studying gas/solid reactions with stepwise control of the process via the injection of a reactive gas, and simplifies adsorption/desorption experiments and studies of catalytic reactions. The valve is entirely controlled via the NETZSCH *Proteus*® software, with no manual intervention necessary.



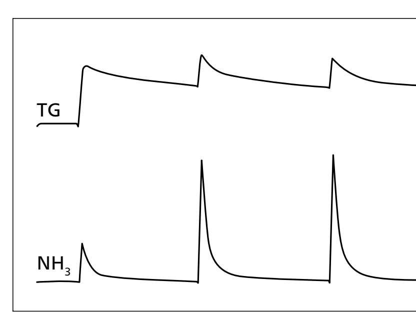
Inert gas
CO_2 pulses for calibration of a carbonate decomposition



Reactive gas (gas-solid reaction)
Reduction of metal oxide by H_2 pulses



Reactive gas (adsorption)
NH_3 adsorption by a zeolite sample



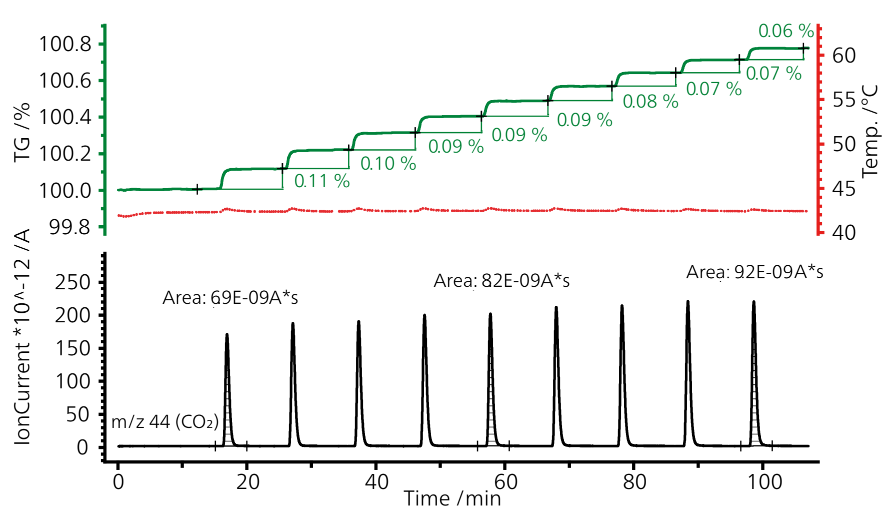
NETZSCH *Pulse*TA® 300

Solid-Gas Reaction of Burned Lime with Carbon Dioxide

In this example, calcium oxide (CaO, burned lime) was treated with defined injections of CO_2 at 43°C. The volume of each injected pulse amounted to 250 µl.

Each gas injection causes a reaction of solid CaO with the reactive CO_2 gas, which can be seen via the stepwise increase in the sample mass. With each consecutive gas uptake, however, the height of the new step reduces.

This reaction may be continued until a plateau in the TGA signal is reached. Simultaneously, the peak area below the MS signal for m/z 44 (referring to CO_2) can be evaluated. The peak area increases as the amount of CaO conversion decreases. The higher the level of saturation with CO_2, the lower the consumption of the pulse gas.



Isothermal treatment of CaO (177.8 mg) on a Pt grid sample carrier at 43°C in a dry argon atmosphere with pulses of 250 µl of CO_2

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