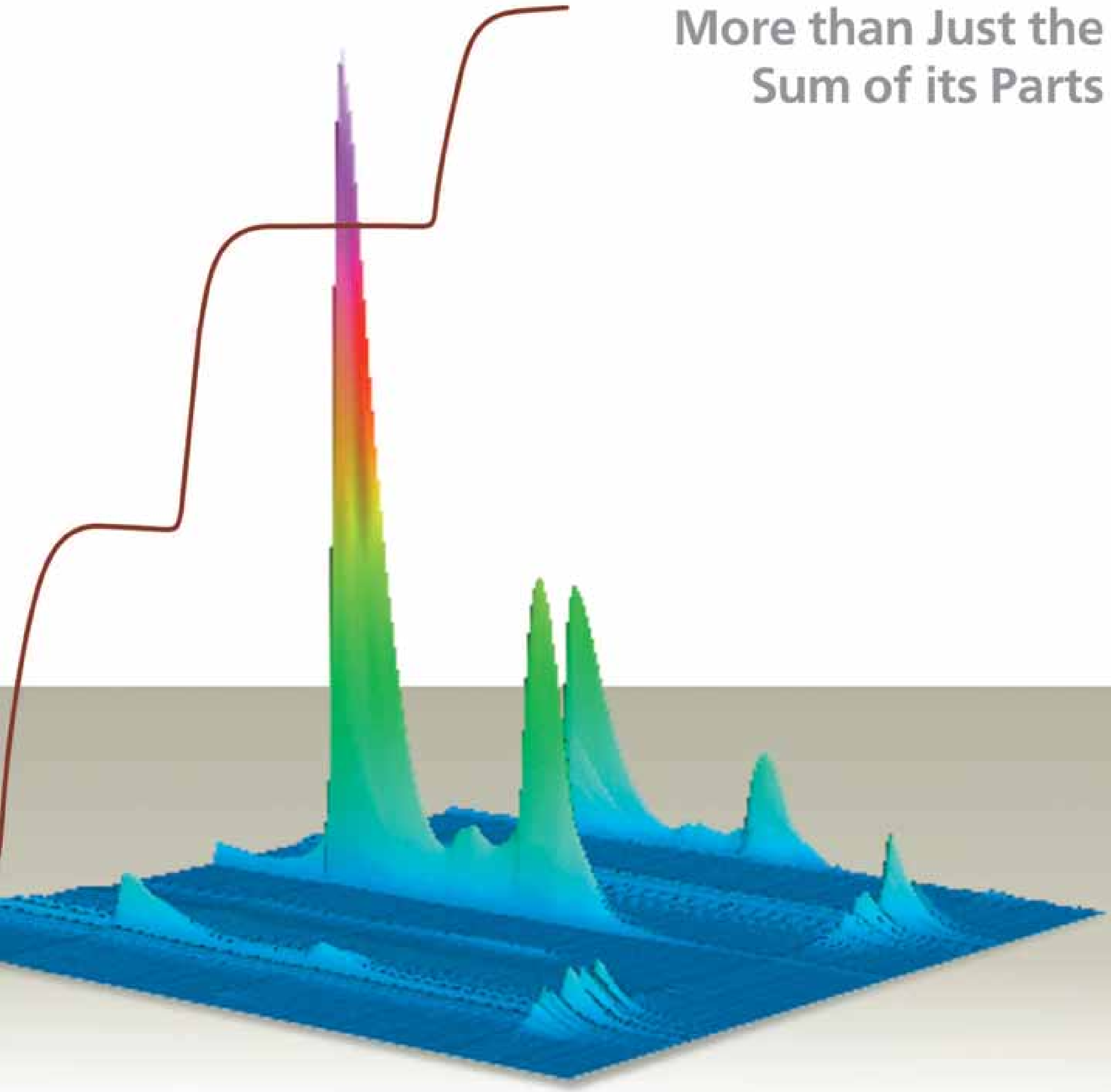
# TG-FTIR

## More than Just the Sum of its Parts



BRUKER

NETZSCH

# Bridging the analytical gap

Thermal analysis provides perfect tools for the characterization of all kinds of organic and inorganic solids or liquids. Thermodynamic transitions, thermal stability, decomposition and chemical reactions are detected and quantified with high accuracy in a broad temperature range. Information about the type and quantity of evolved gases, i.e. details about the chemistry behind the processes under study, are lacking in most experiments, however. The connection of thermal analysis with the powerful infrared spectroscopy for gas analysis bridges the analytical gap.

A NETZSCH thermobalance (TG) is the general basis for couplings. The evolution of volatiles during a heating program is measured by corresponding weight changes down to a level of 0.01% of the sample weight. A purge gas flow carries the volatiles through a short heated transfer line to the gas cell of a BRUKER Fourier-Transform Infrared (FTIR) Spectrometer. All gases with a changing dipole moment are identified by their typical absorption spectrum and complex gas mixtures can be spectroscopically separated.

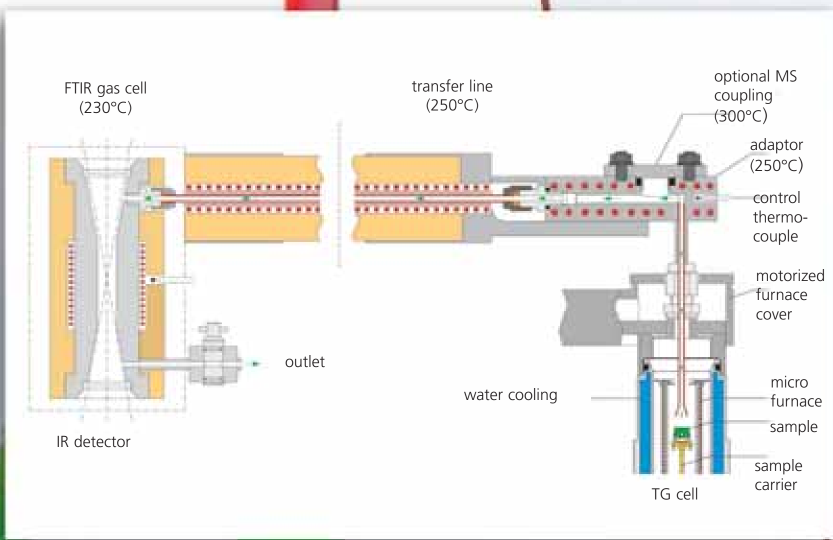
Areas of application:
- composition of materials
- solid-gas reactions
- combustion products
- residual solvent content
- precursor reactions
- evaporation, outgassing

# Optimized TG-FTIR coupling

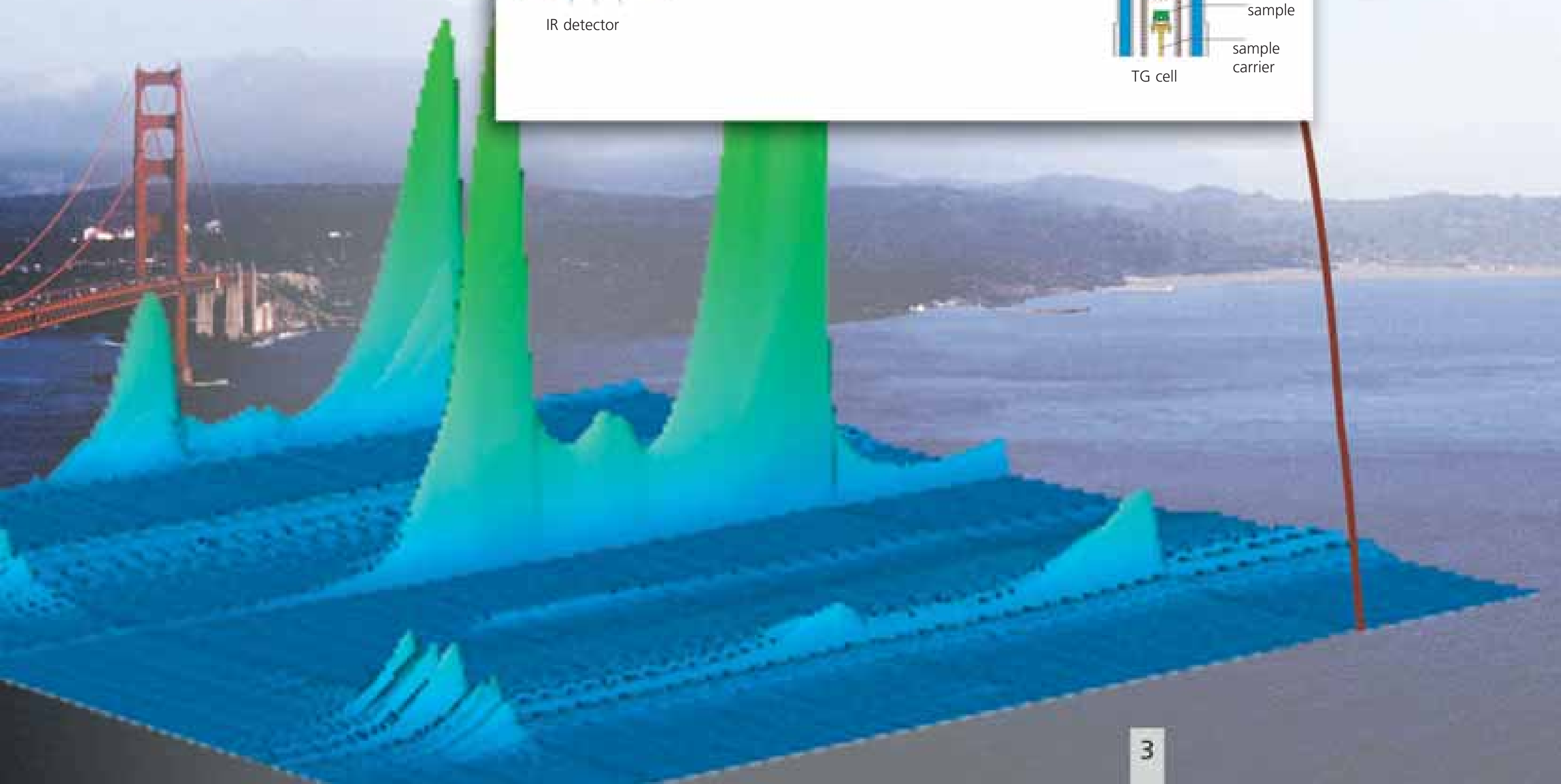
The TG-FTIR coupling designed through the joint efforts of BRUKER and NETZSCH engineers resulted in a real functional unit, both for the hardware and the software solution. An outstanding sensitivity for the gas analysis is provided due to low purge gas flow rates at the thermobalance, a natural, vertical gas flow around the sample, a heated furnace gas outlet, the shortest possible heated transfer line, and a beam-conforming stainless steel gas cell with a sensitive IR detector.

This is supported by a comfortable integration between the *Proteus*® software for thermal analysis and the OPUS software for the FTIR. All information is strongly correlated with temperature and time of the running experiment.

Key features of the coupled TG-FTIR systems
- excellent sensitivity
- high resolution of superimposed effects
- easy cleaning
- complete system vacuum-tight
- easy control from one PC



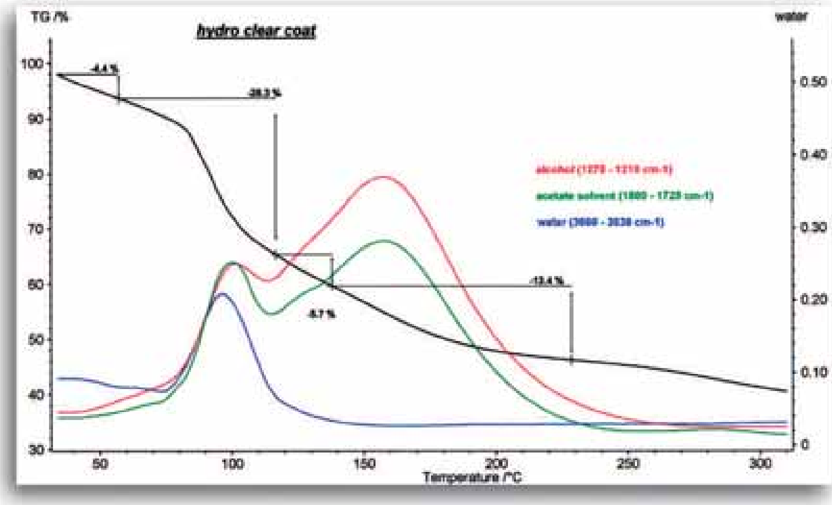

TG-FTIR coupling with heated adapter, transfer line and gas cell



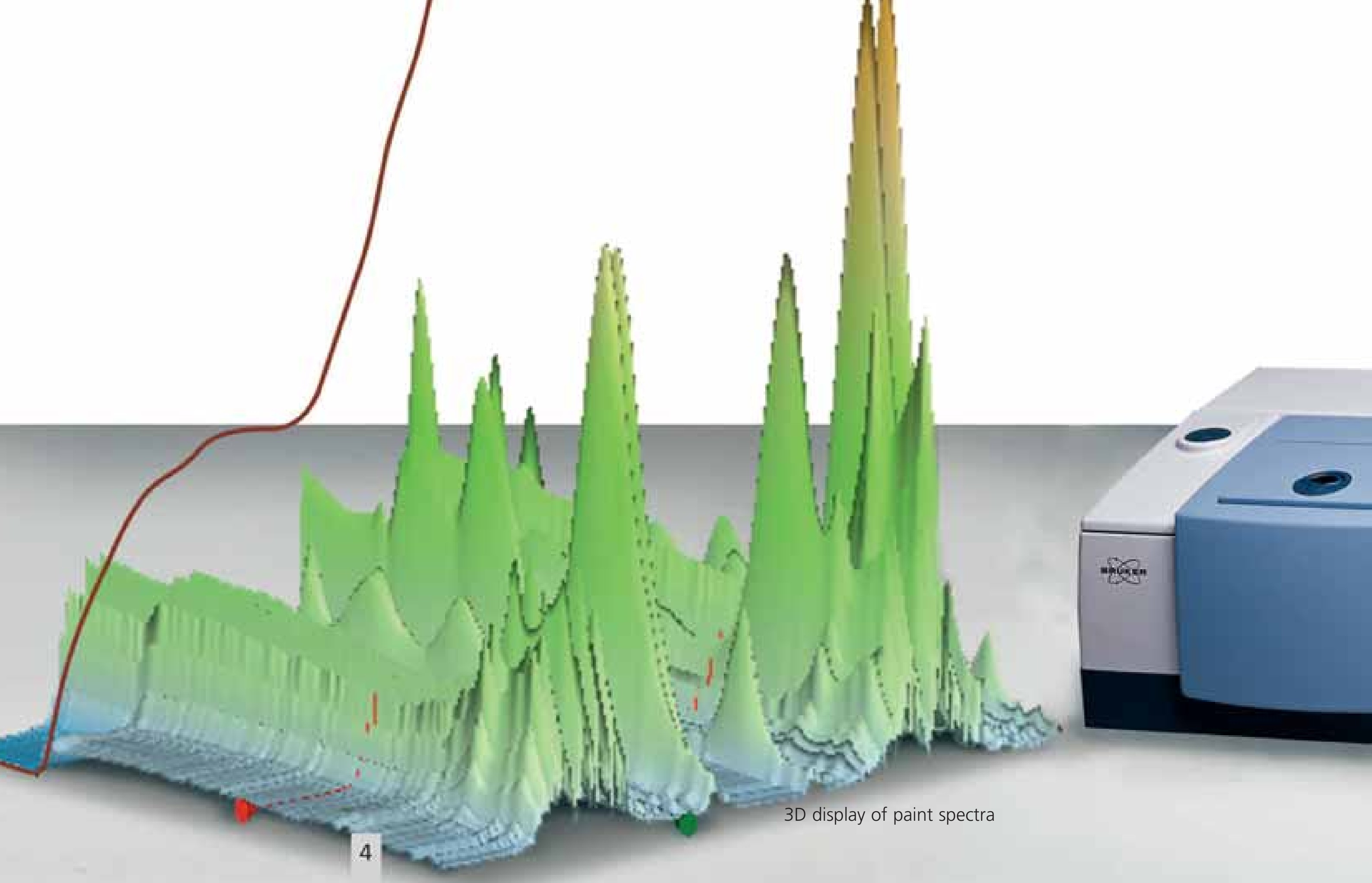
# Application: Curing Powders and Paints

Volatile components in paints are of environmental issue during application. Water-based paints and powder coatings reduce this problem to a great extent.

31.9 mg of a two component hydro clear coat were analyzed in the TG 209 ***F1*** *Iris*® coupled to a TENSOR™ 27 FTIR spectrometer. The sample was heated at a rate of 5 K/min in a nitrogen flow of 45 ml/min up to 300°C. The main weight loss up to 100°C was clearly attributed to the water, but significant contributions come also from hydrocarbons, like alkyl acetates and aliphatic alcohols. The maximum evolution rate for these latter components is shown by the two peaks in the traces at 154°C. During drying of this clear coat, no indication of harmful or toxic volatiles was found.

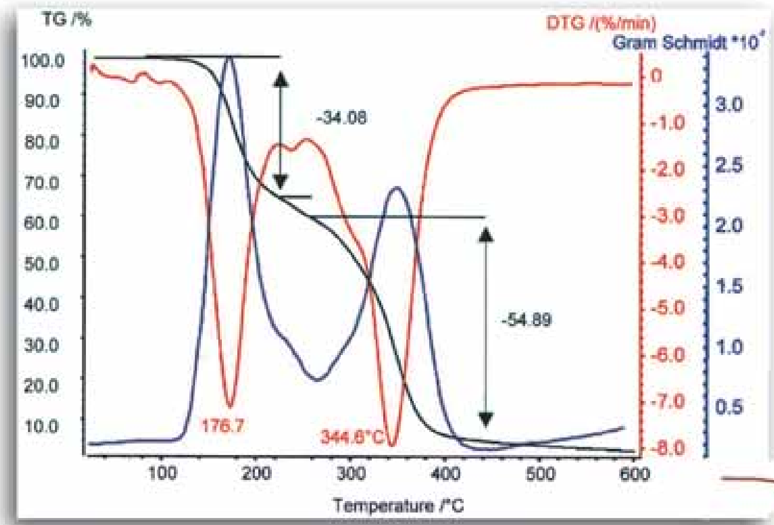


Drying and curing of water based paint

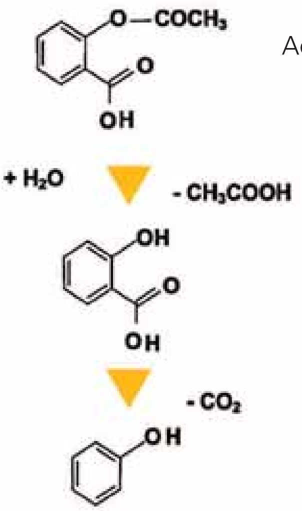

3D display of paint spectra

# Application:
# Medical drug products

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




Thermal decomposition scheme of acetylsalicylic acid



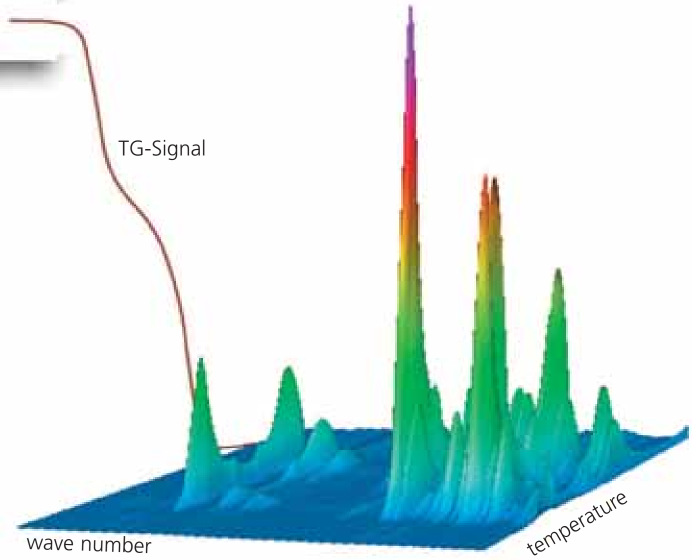

Acetylsalicylic acid (Aspirin®)

Salicylic acid bp. 211°C at 1013 mbar

Phenol bp. 181°C at 1013 mbar

Decomposition scheme of acetylsalicylic acid

A piece of an Aspirin® tablet was heated at a rate of 10 K/min in a nitrogen flow of 45 ml/min up to complete decomposition. The two main steps of weight loss contribute to the evolved gas mixture with mainly acetic acid, salicylic acid, phenol and carbon dioxide. The high boiling components are efficiently transferred through the heated transfer line to the gas cell and clearly detected by FTIR. The reaction and decomposition scheme is shown by the structural formulas.

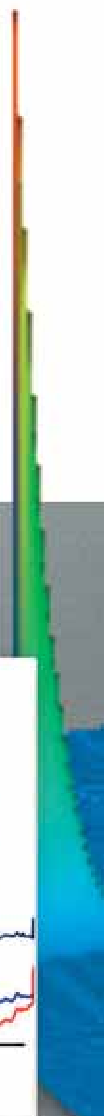


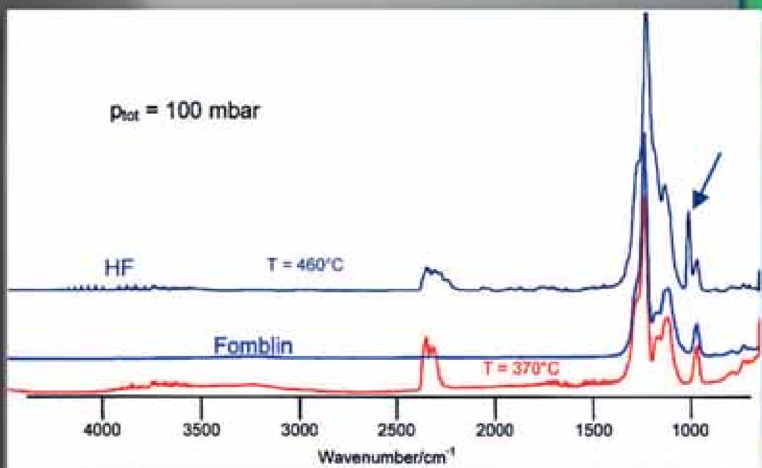


Coupling TG 209 ***F1*** *Iris*® - Tensor™ 27 with external gas cell

# Application: Reduced pressure for high boiling products

The detection of volatile products with boiling temperatures strongly exceeding the heating of the transfer line requires special care. The vacuum-tight design of the BRUKER-NETZSCH coupling system allows working at a reduced pressure. This way, the boiling temperature of volatile samples is also reduced to a range where the passage through the transfer line is without loss. It is possible to detect high boiling plasticizers in polymers and rubbers like Fomblin® in a perfluorinated O-ring, tested below. At a pressure of 100 mbar in the thermobalance and the whole gas path, the plasticizer Fomblin® which evolved from the O-ring was identified at 370°C by comparing with the spectrum for the pure substance. At higher temperatures (460°C), decompostion products of the polymer, like HF and other fragments at the marked absorption bands, are additionally detected.

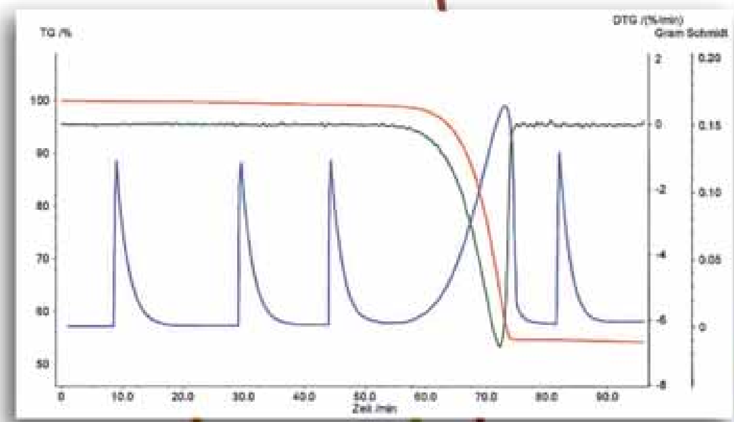





Detection of evolved gases during heating of a perfluorinated rubber at reduced pressure

# Calibration + Quantification

The unique *Pulse*TA® is based on the injection of a specific amount of gas or liquid into the purge gas flow and monitoring of the changes of sample mass, enthalpy and evolved gases.




TG-DTG and Gram-Schmidt plot with 4 calibration peaks and a sample peak for $CO_2$ evolution from $CaCO_3$

*Pulse*TA® offers three principal means of thermoanalytical study, depending on the kind of injected gas or liquid:

### Injection of an inert gas which does not interact with the sample

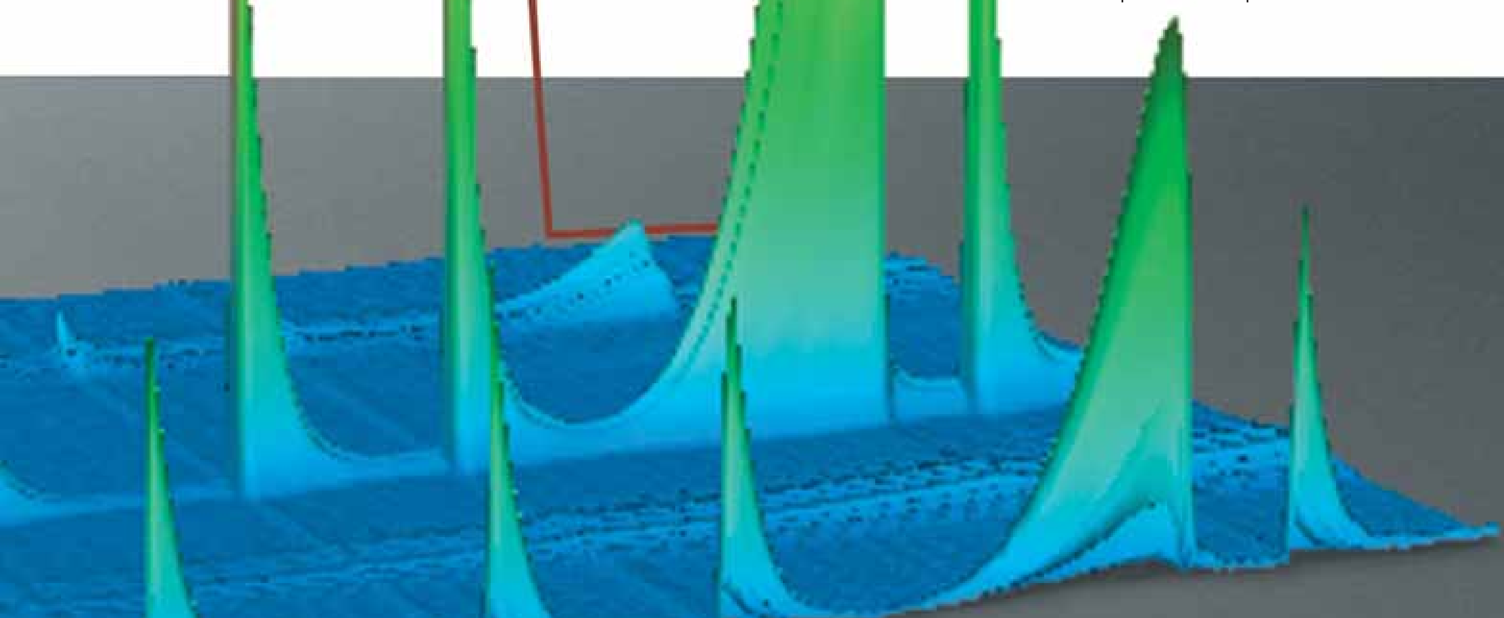
Because the amount of injected gas is known, this mode can be used for a precise calibration of the coupled TG-FTIR for quantitative gas analysis and it increases the sensitivity for detection of volatile contents even below 0.01 %.

### Injection of a gas which chemically reacts with the sample

This mode provides the opportunity of investigating all types of solid-gas reactions at incremental reaction extents (e.g. catalyst research).

### 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 specific temperature.



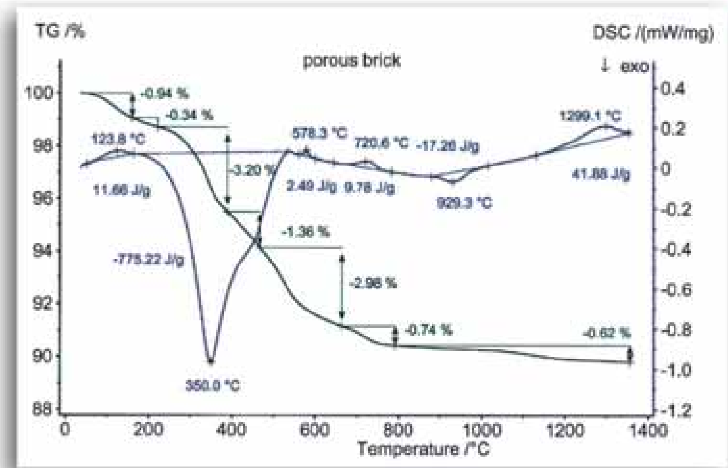
3D plot for $CO_2$ pulses and $CaCO_3$ decomposition

# Application: Building materials

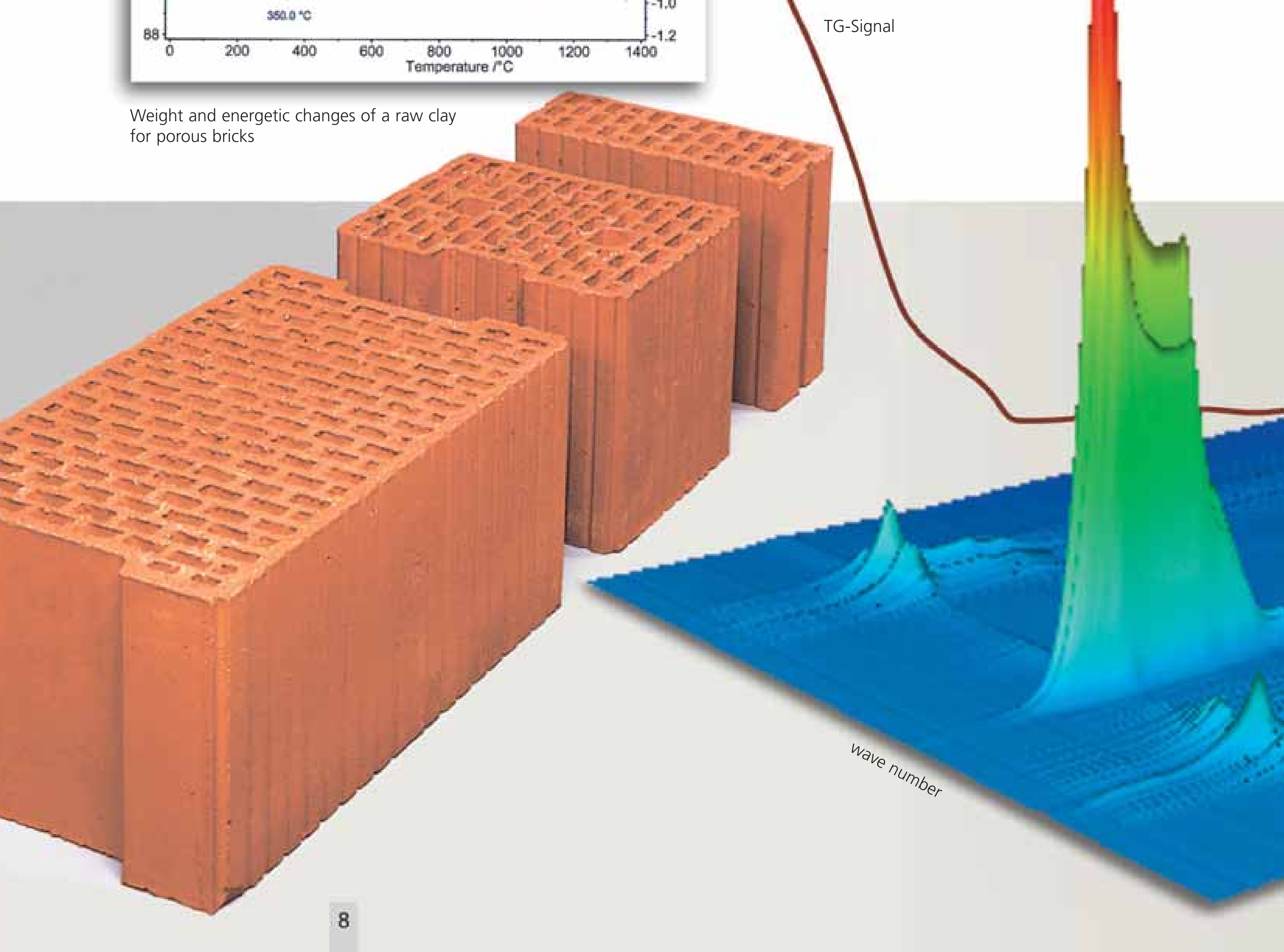
Energy-saving in building requires wall constructions with low thermal conductivity. This is achieved using building bricks with high porosity. Various organic products capable of producing a high volume of voids are mixed into the clay to form the cavities during firing.

The burn-out of the organics in a traditional clay brick is accompanied by high energy release (775 J/g). Water and carbon dioxide are the main volatiles during the binder burn-out, but the FTIR also clearly detects the evolution of HF and $SO_2$ from the clay.

The identification of the emissions allows the optimization of the firing process from economical and ecological viewpoints.



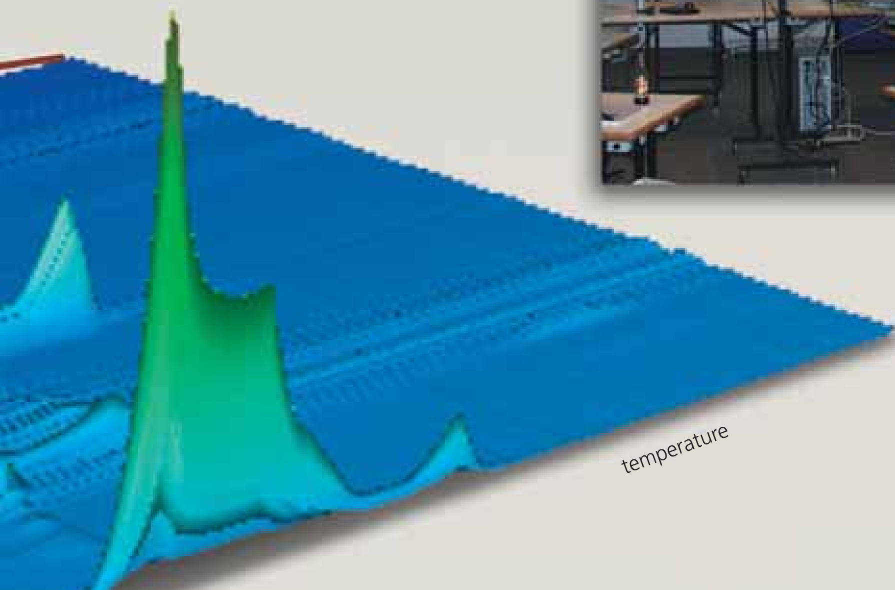

Weight and energetic changes of a raw clay for porous bricks



# Training, Application service, Contract testing

Short start-up time, efficient use of new coupled instruments and helping our customers interpret results are goals of our after-sales support teams. Learn from our experience and that of our users in special training courses and users' meetings, while visiting our well equipped application laboratories, or during hands-on workshops. Enjoy your travel in beautiful regions when visiting BRUKER Optik GmbH in Ettlingen or NETZSCH-Gerätebau GmbH in Selb, Germany.

If an investment in a coupled instrument from BRUKER or from NETZSCH is currently not in your horizon, please allow us to offer you our excellent testing services for all your sampling needs.



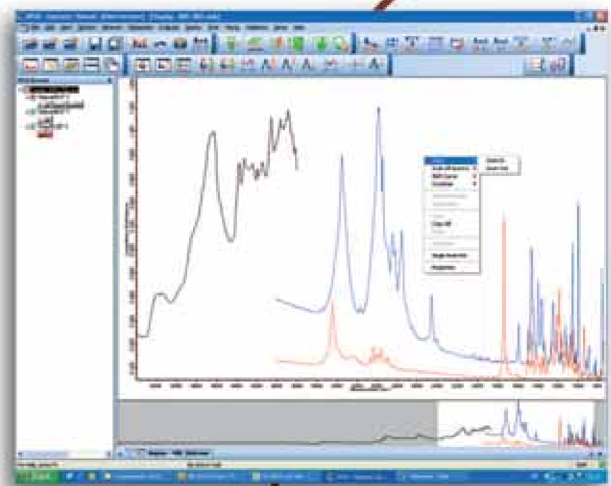

3D plot for gas evolution during brick firing

# Software
# OPUS for comprehensive FTIR measurements & evaluations

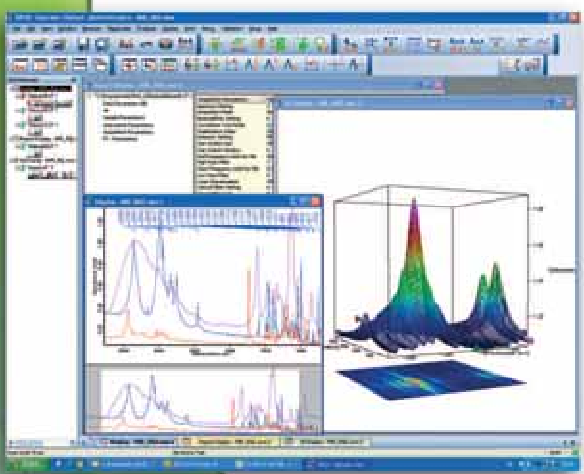
The OPUS user interface offers many features for displaying and managing infrared spectra. Customizable pop-up menus and tool bars allow users to configure the OPUS software to their applications.

The OPUS / CHROM software provides special routines for setting up experiments and performing evaluations for the hyphenated techniques, especially TG-FTIR. Both the integral Gram-Schmidt chromatogram trace and spectral windows traces for interesting absorption bands can be calculated and displayed in real time.

The OPUS / 3D is a package for processing and viewing results obtained with the OPUS / CHROM. It supports 2D and 3D representations of the data together with TG and time data. It also allows 3D files to be created from individual spectra. All manipulations and evaluation functions offered in the OPUS software can be applied to the 3D.
The OPUS / SEARCH package contains an extensive array of search functions in addition to a full featured library editior. The search software can be used with a large set of commercial libraries or create and maintain user own libraries.



screen dump: spectra of paint measurement and evaluation

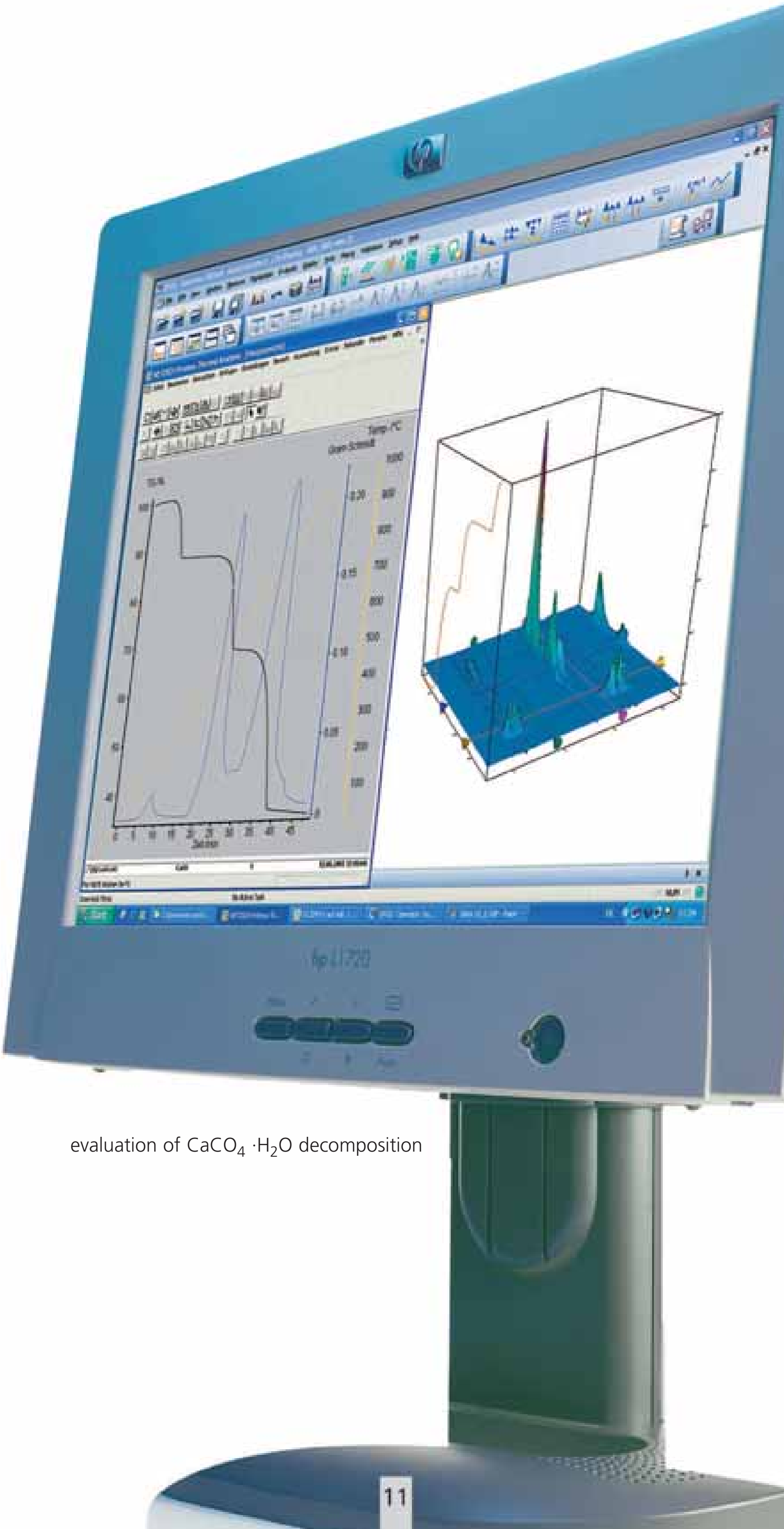


screen dump: multi-window spectra evaluation

# Software
# *Proteus®* for Thermal Analysis
# Integration with OPUS / CHROM

The control of measurements with TG-FTIR instruments is governed by the *Proteus®* software. The user gives the command for data acquisition, once both the OPUS and the *Proteus®* software have had the parameters inputted. The online data collection is simultaneous and synchronized so that later, during evaluation, a precise time and temperature correlation between all the signals from the two coupled analytical systems is guaranteed. The user works with the two software packages on one computer and has all possibilities of evaluating data and displaying results in the OPUS and in the *Proteus®* software according to his preferences.

The integration of the two software packages based on effective data exchange from acquisition to evaluation makes the coupled TG-FTIR a real functional unity.



evaluation of $CaCO_4 \cdot H_2O$ decomposition

Special TA-FTIR features:

- simultaneous TG-DSC/DTA-FTIR instrument control and data storage on the same PC
- combined analysis graphics of TG-DSC/DTA and FTIR signals
- trace calculations with evaluation of characteristic temperatures and peak areas together with TA curves
- Gram-Schmidt plot with temperature and peak area calculation together with TA curves

# Versatility in couplings

Why just coupling FTIR? Many reasons favor the infrared spectroscopy for the analysis of volatile products during thermal analysis on organics, polymers, rubbers, paints, biomaterials, drugs and food. But why not using additionally and even simultaneously a mass spectrometer to get information on all evolved species?

Ask for our well proven solutions for simultaneous TG-FTIR-MS couplings. Or are you looking for state of the art solutions for mass spectrometer couplings and for GC-MS couplings? Let us show and demonstrate you our proposals, based on more than 30 years of experience in the field of coupling gas analysis methods.

BRUKER entered the field of FTIR spectroscopy in 1974. The early instruments set new standards in research FTIR with evacuable optics, high resolution and automatic range change. Since then, the product line has been continuously expanding with instruments suitable for both analytical and research applications with exceptional performance characteristics.

BRUKER led the way establishing FT-Raman as a major new analytical technique in the late 1980s. The recent introduction of dedicated FT Near IR systems for QA/QC has enhanced the reputation of BRUKER Optics as a leading supplier of FTIR and FT-Raman instrumentation.

BRUKER Optics Inc. has R&D and manfacturing centers in Germany and in the USA, supporting technical centers and offices throughout Europe, North and South America and Asia.

BRUKER OPTIK GmbH
Rudolf-Plank-Str. 27
76275 Ettlingen
Germany
Phone: +49 7243 504-600
Fax: +49 7243 504-698
E-mail: info@brukeroptics.de
www.brukeroptics.de

NETZSCH-Gerätebau GmbH offers a complete product line for the thermoanalytical and thermophysical characterization of materials within the scope of research, development and quality control. DSC, DTA, TG, STA, DMA, DEA, TMA, dilatometers and instruments for thermal conductivity and diffusivity form the core of the production program developed since 1953. We provide instruments for measurements in the extremely broad temperature range from -260 to 2800°C, various couplings for FTIR and mass spectrometers, as well as a host of accessories for applications in research, development and quality assurance. Our well-equipped applications laboratories around the world are at your service for consultation or on a contract basis to demonstrate the excellent capabilities of our instruments.

NETZSCH-Gerätebau GmbH
Wittelsbacherstr. 42
95100 Selb
Germany
Phone: +49 9287 881-0
Fax: +49 9287 881-505
E-mail: at@netzsch.com
www.netzsch.com

NGB · TG-FTIR · E · 1000 · 0508 · MMM