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YEARS ANNIVERSARY

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DSC 300 Caliris

NETZSCH

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Dear Reader:

NETZSCH-Gerätebau GmbH was founded 60 years ago – so this year, we have a nice opportunity to also look back. Our creative minds have given birth to many wonderful activities for this occasion which will accompany us throughout 2022. I was particularly impressed by our monthly competition, in which we invite our customers to submit entries for the oldest instruments in operation for each of the various methods of thermal analysis. We've received numerous submissions from customers who have been using their instruments for over 30 years!

It has always been our policy to not discontinue instrument support or even discontinue service – even a 30-year-old NETZSCH instrument is a good instrument and will reliably do what it has already done for decades. Now that sustainability is becoming increasingly important, we are happy to continue this more-than-contemporary company policy. On page 21, you will find a link to our monthly competition and, starting on page 8, a brief company history.

Now, however, I would like to look ahead: A few days ago, we unveiled our new DSC 300 *Caliris*[®] to the public. With this instrument, we aim to set new standards in thermal analysis. Read our exciting cover story and see for yourself how many compelling benefits this instrument offers.

In line with the mega trend of digitalization, we have networked with renowned partners and founded LabV[®]. Data flows in the laboratory can now be intelligently managed across all devices – in a cloud-based and traceable fashion.

In the 2nd part of our series "Next-Generation Energy Materials", we look at the development of new materials for this increasingly important field and demonstrate how our STA 449 **F1** Jupiter[®], coupled to a QMS Aëolos[®] Quadro, can contribute to success here.

It's been a pleasure to witness the development of our relatively young pillar: From a single source, we offer fire testing compliant with a variety of international standards. Our article on the TBB 913 describes how important such tests are for determining fire behavior and classifying fire classes.

The article under PRECISE PRACTICE highlights the determination of oxygen content in a thermobalance.

Our customer service has also steadily developed over the last 60 years and stands by your side with proficient assistance. With the memories presented by our customer service manager from China, we would also like to make you smile.

The entire NETZSCH Group has been dedicated to the red-hot topic of batteries for some time now. Read more about this on page 26.

Last but not least, I would like to draw your attention to the new application book on thermal analysis methods for metals and alloys.

We'll stay in touch – live or virtually! How I hope you enjoy reading this edition of onset.

Yours truly,

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Dr. Thomas Denner Head of Business Unit Analyzing & Testing Managing Director



Sharing – Newly Interpreted with the Innovative DSC 300 *Caliris*®

Dr. Gabriele Kaiser, Business Field Management for Pharmacy, Cosmetics & Food



Fig. 1. DSC 300 Caliris®

Maximum Performance with the DSC 300 *Caliris® Supreme* at Lower Costs

Excellent research requires excellent instruments – be it in the academic world or in industry. Sometimes, however, budget does not allow for the realization of each and every wish. The new DSC 300 *Caliris® Supreme* by NETZSCH (see figure 1) offers a completely new approach here. Thanks to its modular design, the base unit can be equipped with different modules. One module comprises a furnace sensor unit and can be changed out by the operator easily in just a few minutes. This allows for different application fields to be covered with only one electronic system and one automatic sample changer, thus saving costs and resources.

Ideal for Institutes and Research Centers

Currently, three modules are available: the high-performance module, H, with the widest temperature range on the market for low-temperature DSCs; the polymer module, P, which can realize very high heating and cooling rates; and the robust standard module, S.

As shown in figure 2, modules belonging to individual project partners or different institutes can be connected consecutively along with the respective

cooling system to a base unit placed at a central location. Capacity bottlenecks are never a problem, as the optional Automatic Sample Changer (ASC) with its 192 positions for sample crucibles offers space enough to measure the accumulated samples over the course of several days or a weekend.



Fig. 2. Schematics of a possible collaborative project using a DSC 300 *Caliris® Supreme*; all project partners acess the same base unit.

DSC 300 Caliris®

Future-Proof

Even if an organization's focus of research should change over time, users of the DSC 300 *Caliris® Supreme* will always be on the safe side. By purchasing a new module suited for the upcoming task, their system can be quickly and easily customized to any changed conditions – at a much lower cost than that of purchasing new equipment.

All Important Information at a Glance

Information about the status of the instrument or the measurement progress can be viewed on a colored LED bar. This makes it possible to tell from across the room whether the DSC is still measuring, for example, or is ready for a new measurement.

The integrated, colored touch display can be used not only to start measurements, but also to monitor their progress or view the evaluated curves with all results. The latter is possible if certain measurement methods are employed, including automatic evaluation (= AutoEvaluation).

Uncompromising Quality in Hardware and Software

In addition to the hardware, the software is also a decisive component in the analyzer's performance. Functionality, reliability and usability are some of the keywords here. The 9th generation of the NETZSCH *Proteus*[®] software, in combination with the DSC 300 *Caliris*[®], goes far beyond fulfilling its purely functional purpose as measurement and evaluation software. Some features of the 8th generation, such as *AutoEvaluation* or *Identify* (= database comparison for material identification), have been refined and expanded upon by additional features such as individually configurable and storable workplaces and the *Proteus*[®] *Search Engine*.

Database Functionality without Expensive Licenses

The *Proteus*[®] Search Engine offers the option to search for measurements or evaluation states by

applying different filter criteria. This can be the name, a time period, or a specific result – be it the glass transition temperature, peak temperature, extrapolated temperature or enthalpy. Within minutes, the user can get an overview of such issues as which samples meet certain specifications within a certain time period. By clicking on the respective measurement in the results list, the associated measurement graph can be displayed.

Optimum Illumination of the Measuring Cell

The cell illumination of the DSC 300 *Caliris*[®] makes manual insertion of sample crucibles into the DSC cell considerably easier (see figure 3). When opening the furnace lid, an LED bar is automatically switched on; then off again when the furnace is closed. Crucibles can thus be precisely positioned on the sensor even under unfavorable ambient lighting conditions.



Fig.3. Integrated light strip of the H module illuminates the measuring

Too Beautiful to Share? Configure Your Own DSC 300 *Caliris® Select!*

The DSC 300 *Caliris*[®] *Select* is a traditional DSC instrument, consisting of electronics, a module plus cooling, software and possibly an automatic sample changer (ASC). With this version, customers decide already at the time of purchase which module is best suited to their particular application. The equipment of the instrument can be expanded in many ways through options. This makes the DSC 300 *Caliris*[®] *Select* ideally suited for investigations within the scope of research projects, as well as for service or routine tasks.

DSC 300 *Caliris*®

Perfect Lips



The main ingredients of lipsticks are oils and waxes, supplemented by fats, color pigments and possibly stabilizers. The more oil a lipstick contains, the softer and more nourishing it is. The challenge is to optimize the formulations so that the lipstick applies well, but still stays on the lips for a long time. The result of these efforts is usually blends with complex DSC profiles like in figure 4.

In this case, a commercial lipstick of cherry red color was subjected to several heating/cooling cycles at different heating rates. Figure 4 depicts a comparison of each 2nd heating, recorded at 1 K/min (black) and 5 K/min (red), respectively.

The thermal behavior of the two samples is largely similar. The duration of the controlled cooling does not seem to be sufficient in either case for all components of the mixture to crystallize entirely. The result is a broad exothermic effect with a peak temperature around -40°C during heating – known as post-crystallization. Significant endothermic effects associated with the melting of various ingredients occur at -14/-16°C (peak temperature) and in the temperature range between approx. 30°C/35°C and 80°C.

The slight differences in the peak temperatures are due to the different heating rates: The higher the heating rate, the more the peak temperature is shifted to higher values.

Remarkable, however, is the temperature range between approx. -15°C and 30°C. While two exothermic effects and one endothermic one can be seen in the black DSC curve (heating rate 5 K/min), the red DSC curve shows only one exothermic and one endothermic effect; the latter is additionally shifted by more than 10 K as compared to the corresponding effect in the black curve.

(Post)crystallizations are kinetically controlled processes and can therefore shift to different temperature values at different heating rates.

At higher heating rates (like in this case, 5 K/min), effects in close proximity are often superimposed, as also occurs with the exothermic phase transition with a peak temperature of -13°C. This is one of the reasons why it is advisable to vary the heating rate when investigating materials that are prone to structural changes.



Fig. 4. DSC measurements on a commercial lipstick at different heating rates; sample masses: 15.3 and 15.7 mg, closed Al crucible, N₂ atmosphere, S module. For a better overview, the DSC curves shown are adjusted in size and shifted with relation to each other on the y-axis.

The new DSC 300 Caliris® Supreme and Select – an offering covering an extremely wide range of applications. Flexible, accurate, precise and reliable – the very definition of Proven Excellence.

For more information, please visit <u>www.netzsch.com/caliris</u>

LabV[®] – Laboratory Software That Frees Up Time!

Dana Seidel, Project Innovation Manager at NEDGEX

In cooperation with renowned development partners, we have created a customer-centric software that reduces manual data management in the laboratory and gives laboratory technicians more time for what is important: working efficiently with the data.

LabV[®] intelligently links test equipment with measurement data and simplifies organization.

The laboratory software offers a modern and intuitive user interface that ensures the ability to carefully manage incoming measurement and test data in a central location, regardless of the device or manufacturer.

The huge advantages of SaaS (Software as a Service) include the enabling of access to digitalization and the guarantee of full networking and traceable data management.

LabV[®] allows for selected results to be depicted visually, as well as for trend analyses and the automatic generation of test reports.

Thanks to our intelligent process, any test instrument that can export a csv/sql file can be easily connected to LabV[®]. This makes project management and data processing efficient and creates comprehensive digitization in the laboratory.

The new, smart laboratory software can run with *Proteus*[®] version 6.0 and higher.

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Fig. 2. LabV[®] demo version screenshot

The following NETZSCH instruments are already connected to the software and thus, 'LabV[®]-primed`:

- NETZSCH DSC 300 Caliris[®]
- NETZSCH TG 209 F1 Libra®
- NETZSCH STA 449 F3 Jupiter[®]

LabV[®] is hosted in the German AWS data center in Frankfurt. All data are encrypted by TLS during transmission, and using the 256-bit Advanced Encryption Standard (AES-256) while at rest. LabV[®] and all AWS services used are fully compliant with EU DSGVO guidelines.

AWS is certified in accordance with ISO/IEC 27001:2013, 27017:2015, 27018:2019, ISO/IEC 9001:2015 and CSA STAR CCM v3.0.1.

Dynamics

Interested in a demonstration? Contact us at k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k type://labv.io k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at k but here information on LabV* can be found at

The History of NETZSCH-Gerätebau GmbH — 60 Years Along the Road of Success

Dr. Thomas Denner and Dr. Jürgen Blumm, Management of NETZSCH-Gerätebau GmbH



Fig. 1. Company headquarters of NETZSCH-Gerätebau GmbH – 60 years ago and today

How It All Started ...

1962 marked the beginning of the success story of NETZSCH-Gerätebau GmbH at the Selb site (figure 1). Within the past 60 years, we have become one of the world's leading manufacturers for thermal analysis. We are proud of our employees who have contributed to this success with an incredible amount of commitment and willpower. Thank you!

And we would like to thank you, our customers and partners, for the trust-filled and successful collaboration. Together, we stand for quality, professionalism, innovation and sustainability and will continue to do so in the decades to come.

Numbers – Data – Facts



Together with you, we would like to duly celebrate our company's anniversary in 2022. Already since January, our campaign has been running under https://ta-netzsch.com/category/60-years-of-ngb with a view behind the scenes. Each month, we are presenting one of our analytical instruments and its development over the past decades. In January, we started with the dilatometer, one of the oldest instruments in our company history. This was followed by simultaneous thermal analysis, our coupling systems, thermogravimetry and in May, differential scanning calorimetry (DSC), the most frequently used thermal analysis method. As part of these campaigns, we ask every month: Who has the oldest analytical instrument still in use? Four winners have already been chosen, and we are proud that so many analyzers from the 1970s are still in use today, delivering reliable measurement results.

A Look at the History

As early as the 1950s, complete assembly lines for the manufacture of porcelain or ceramic products were set up under the direction of the Netzsch brothers. In the course of supplying complete production facilities for the fine ceramics industry, customers requested to be able to also purchase associated testing or laboratory equipment. That was the reason for the decision to develop and manufacture special instruments for setting up ceramic laboratories.

60 Years of NGB

The development of such equipment initially started on a small scale: Ideas were implemented in the apprentice workshop of the former Maschinenfabrik Gebrüder NETZSCH in the form of experimental apparatuses. In order to intensify activities around development, production and sales in the "test instruments" department, NETZSCH-Gerätebau GmbH was founded on June 27, 1962, with its head office in Selb. Dilatometry (DIL, see figure 2), differential thermal analysis (DTA) and thermogravimetry (TGA) formed the three pillars in the early years of thermal analysis.



Fig. 2. One of the first dilatometers from 1955

Evolution of Our Company Logos Over Time

Our logos have also evolved over the decades. In the early days, the company name and our business unit's profile still played a dominant role in the logo. In addition, a sketch was often used based on a DTA curve of kaolin. Today, we have a logo that is identical across all business units of the NETZSCH Group as a promise to our customers: Proven Excellence stands for the combination of state-of-the-art technology, decades of experience and a focus on our customers' needs.



Fig. 3. Development of our company logos from 1962 to 2022

Proven Excellence – 1962 until Today

"Proven Excellence – we have been living this philosophy for decades. At the beginning of the 1970s, Dr. Wolf-Dieter Emmerich (figure 4), who shaped the company as Managing Director for more than 30 years, established a team that has always operated in close proximity with current research topics.

With his management team consisting of Martin Schmidt, Gerhardt Bräuer, Karl Bayreuther and Rudolf Lohse, Dr. Emmerich always kept in touch with universities and research institutions. Together, they laid many technological milestones. Customer proximity always took center stage. The establishment of subsidiaries and branch offices with service and support staff all over the world constituted the foundation for optimum user support," explains Dr. Jürgen Blumm.



Fig. 4. Dr. Wolf-Dieter Emmerich: Managing Director of NETZSCH-Gerätebau GmbH from 1974 to 2005

60 Years of NGB



Customer Orientation and Process Optimization

Our positioning today is: "We deliver tailored solutions going beyond measurement data and provide insight with smart approaches!" We were never of the opinion that a "One Size Fits All" approach is a good answer to the multiple challenges in materials analysis. In fact, we have always accepted the challenge of providing our customers with a solution that fits their requirements. Today, with up to 400,000 possible instrument configurations, we manage to find exactly the solution that is tailored to the individual customer's requirements.

In line with the steady extension of our headquarters, the development of processes has been and will continue to be implemented. Departments have moved closer together, distances have become shorter, personnel deployment has become more flexible and communication is now faster and better.

Accordingly, our logistics and production processes are now streamlined and efficient, enabling us to respond flexibly to our customers' needs and always deliver the highest quality.

Parallel to the extension of the Selb site (figure 6), numerous foreign subsidiaries (figure 5) have been modernized into state-of-the-art companies over the decades and further acquisitions have been made.

Fig. 5. The foreign branches of Analyzing & Testing Business Units in North America, China, Poland, Czech Republic, Korea and Japan

Global Player in the Fields of Thermal Analysis, Rheology and Fire Testing

"When I started working at NETZSCH in 2004, the employees made a particularly positive impression on me. On the one hand, I sensed an enormous wealth of experience in their minds – I still chanced to get to know some of the colleagues from the company's very first days – and on the other hand, there was still a great willingness to venture toward new shores. The combination of pride in what has been achieved and the will for continuous further development is still in the air today", says Managing Director Dr. Thomas Denner, describing his beginnings at NETZSCH.



Thanks to this irrepressible will to constantly develop and try out new things, NETZSCH-Gerätebau has turned into a global player in the field of thermal analysis over the past decades. In order to complete the portfolio and offer the customer a fullservice package, the product range was expanded in 2020 by rheometers and fire testing systems.

Fig 4. NGB building sections 1 to 5: Section 1: 1969, section 2: 1980, section 3: 1987, section 4: 2011, section 5: 2017

60 Years of NGB

Best Performance in All Areas

In the past 60 years, we have developed numerous products and methods and have driven innovation. At NETZSCH-Gerätebau, there are currently 266 active patents and 334 active trademarks.

Along with the quality and performance of our measurement instruments, customer support has also continuously evolved. Today, we offer unique service and applications support – worldwide and first hand. We have more than 40 scientists employed in our applications laboratories around the world available to you before, during and after instrument investment. This means that we can provide you with fast and comprehensive advice, even on complex issues.

The range of training courses – online, via our NOA learning platform or on site – is continuously growing. NETZSCH-Gerätebau also offers an extensive portfolio of free webinars, current application literature and scientific articles.

Outstanding technologies in line with the best possible support have always been and continue to be the key to success at NETZSCH-Gerätebau GmbH.

... And Successes Must Be Celebrated!

NETZSCH has always loved to celebrate! Be it at the annual Wiesenfest in Selb, at Christmas or summer parties or the ever-popular "Scheunenfest" (barn party) with a barbecue with grilled suckling. Especially this year, we will be celebrating the commitment of our coworkers who have accompanied us over the past 60 years and shaped the company with their know-how, adaptability and passion.

And we will be celebrating with you, our customers, who meet us at eye level. With you, we build up a collaboration and an appreciative relationship – and to you, we are honored to prove our reliability again and again every day.

Visit Our In-House-Exhibition

On the occasion of our anniversary, we would like to invite all interested parties to an in-house exhibition*



Fig. 6. View into the Production Department of the most recent construction: Short distances and fast communication between administraton, development and production.

for the first time at our premises on Monday, July 4, 2022. Details and registration information can be found on <u>www.netzsch.com/hausmesse</u>.

On Saturday, July 2, the Open House* at NETZSCH-Gerätebau will take place. Along with guided tours of the company and a physics show, other exciting highlights await you.

Let's toast to 60 years of NETZSCH-Gerätebau! We are looking forward to welcoming you and to many more years together of thermal analysis, rheology and fire testing.

Yours truly, Dr. Thomas Denner (on the left) and Dr. Jürgen Blumm (on the right), Managing Directors



Next-Generation Energy Materials

Benchmarking Storage and Conversion Properties Using Thermal Analysis (Part 2)

Andrew Gillen, NETZSCH Australia Pty Ltd., and Dr. Michael Schöneich, NGB Applications Laboratory



Hyphenated Thermal Analysis: Enabling New Insights into Thermally-Induced Chemical Reactions

In part 1 of this feature, we discussed the flexibility of the NETZSCH STA 449 *Jupiter*[®] Simultaneous Thermal Analyzer Series, including its ability to operate under oxidizing, reducing, inert, corrosive and humid atmospheres.

Research groups developing materials and processes for energy storage applications require an intimate understanding of chemical reaction mechanisms in order to advance the current state-of-the-art. Combining thermal analysis with analytical gas detection techniques brings many advantages; ultimately the ability to identify reaction species and elucidate thermally-induced reaction mechanisms. This approach, more broadly known as 'evolved gas analysis' or 'hyphenated thermal analysis', was first reported in the early 1950s [1-3]. Since then, hardware and software technologies have advanced significantly and the approach is now accepted as a mainstream materials characterization technique.

When a thermobalance is coupled to a suitable mass spectrometer (TGA/STA-MS), the 'evolved gas analyzer' can be conveniently used as a screening tool for

studying solid-gas reactions. For example, in catalysis research, Temperature Programmed Reduction (TPR), Temperature Programmed Oxidation (TPO) and Temperature Programmed Desorption (TPD) experiments [4] can all be realized with a hyphenated thermal analyzer.

Today, mass spectrometry is the most commonly used evolved gas analysis method used in combination with thermal analyzers. Mass spectrometers offer excellent sensitivity, are relatively compact and provide continuous sampling throughout the course of thermal analysis measurements. The usually built-in quadrupole mass spectrometers are composed of a mass filter, an electron impact ion source and an ion detector, and must operate under high vacuum, to ensure the mean free path length of ionized species is sufficiently high to reach the detector unimpeded. For this reason, an interface is required for the coupling of a thermal analyzer, operating at atmospheric pressure, to the mass spectrometer.

Using a capillary tube of small internal diameter, a single-step pressure reduction can be realized within a timeframe of ~100 ms, which connects the gas outlet on the furnace of the thermobalance with the gas inlet on the mass spectrometer. The pressure drops from atmospheric pressure to high vacuum in one continuous step (Figure 1). By measuring the mass numbers (m/z), conclusions can be drawn on the composition of the evolved gases, and quantification of the reaction products is also possible.



Fig. 1. The capillary interface – single-stage pressure reduction step between a thermal analyzer and a mass spectrometer

Another important factor in the interface design is mitigating the condensation of volatile components between thermal analyzer and mass spectrometer. Without sufficient heating of the interface, high boiling point species condense prior to reaching the mass spectrometer, causing blockages of the coupling interface. A capillary interface can typically be heated up to a maximum temperature of 300 - 350°C. For high

QMS Aëolos® Quadro____

boiling point decomposition products, volatile inorganics (e.g., molten salts [5]) and analysis of metal vapors, a different coupling solution is required. The 'Skimmer' interface, by its innovative design, makes it possible for the entire coupling interface to be heated up to as high as 1950°C (depending on configuration). The skimmer interface achieves a pressure reduction from thermobalance to mass spectrometer over a very short distance via two stages (figure 2). Although more complex and more expensive than a capillary interface, the Skimmer coupling makes it possible to conduct evolved gas analysis on many materials that cannot be analyzed using a capillary coupling.

QMS *Aëolos*[®] *Quadro*: The Seamless Mass Spectrometer Coupling for Thermal Analysis

NETZSCH Analyzing and Testing has over 40 years experience [6-8] with mass spectrometer couplings for thermal analyzers, offering both capillary and Skimmer [9] solutions to meet the needs of energy materials



pump 10³ mbar 1 mbar 10⁵ mbar pump Step 1 Step 2 Thermobalance Interface Mass Spectrometer

Fig. 2. The *SKIMMER* interface – double-stage pressure reduction step between a thermal analyzer and a mass spectrometer

research and development. NETZSCH thermal analysis devices were developed from the outset [5, 6] with coupling extensions in mind. The gas transport path, which starts at the furnace outlet and progresses via the heated adapter and capillary up to the QMS inlet, has been studied and optimized at every new phase of development. Today, any gas loss caused by condensation at cold spots has been almost completely eliminated. In addition, the top-loading design of NETZSCH thermobalances ensures that only very low carrier gas flow rates are required for complete gas transfer.

Hence, the STA 449 *Jupiter*[®]-QMS 403 *Aëolos*[®] *Quadro* solution (figure 3), by design, guarantees minimal dilution of the volatile sample products released and therefore highest detection sensitivity.

The 403 Aëolos® Quadro quadrupole mass spectrometer is a state-of-the-art compact mass spectrometer with a heated capillary inlet system for routine analysis of gases and, in particular, volatile decomposition products of thermal analysis. The system is available in 300 amu and 512 amu mass ranges and uses two yttrium oxide coated iridium cathodes, making it possible to operate the system even in oxidizing atmospheres.

The 403 Aëolos® Quadro is optimized for coupling to thermogravimetric analyzers (TGAs) and simultaneous thermal analyzers (STAs). Uniquely, STA/TGA-MS measurements can be controlled completely with the NETZSCH *Proteus*® software.

Fig. 3. NETZSCH STA 449 **F3** Jupiter® coupled to the QMS 403 Aëolos® Quadro mass spectrometer

QMS Aëolos® Quadro

The *Proteus*[®] software combines the measurement and analysis software of the two coupled methods into a single software application for both control and data acquisition (figure 4).

Example: Hydrogen Production Using the Thermochemical Water Splitting Route

Thermochemical water splitting describes the process of hydrogen production at high temperatures (500°C to 2000°C) via a series of chemical reactions. The chemicals produced during the process are reused in each cycle, creating a closed loop system utilizing only water, hydrogen and oxygen (figure 5). Thus, thermochemical hydrogen production represents an environmentally friendly alternative to hydrogen production systems based on fossil fuels [10].

To investigate the thermochemical water splitting with LSC20 (La_{0.8}Sr_{0.2} CoO₃), thermogravimetric measurements (TGA) were carried out with a NETZSCH STA 449 **F3** Jupiter[®]. The thermal analyzer was coupled with a quadrupole mass spectrometer NETZSCH QMS Aëolos[®] Quadro. A summary of the measurement conditions can be found in table 1.

In the first step of the investigation (refer to figure 6), the reduction of LSC20 was carried out in a reducing atmosphere (4% H_2 in argon). The sample material shows a pronounced mass loss of 11.0%.

Using the mass spectrometer coupling, the consumption of hydrogen (mass number 2) with simultaneous water release (mass







Fig. 5. Schematic of the thermochemical water splitting process by means of a metal oxide used for hydrogen production in the energy sector

Table 1. Measurement parameters

Parameter	Measurement Conditions (LSC20)
Instrument	STA 449 F3 Jupiter [®] + QMS 403 Aëolos [®] Quadro with water-vapor furnace and generator
Sample carrier	TGA type S
Crucible	TGA slip-on plate made of Al_2O_3 with 17-mm diameter
Sample mass	215.46 mg (powder)
Measurement program	RT 1200°C, 15 K/min, forming gas* 90 min isotherm @ 1200°C, forming gas* 1200°C 600°C, 15 K/min, forming gas* 30 min isotherm @ 600°C, in argon 180 min isotherm @ 600°C, 33% H ₂ O in argon 30 min isotherm @ 600°C, in argon

*4% H, in argon

QMS Aëolos® Quadro

number 18) can be clearly observed. The actual thermochemical water splitting takes place in the second part of the study. For this purpose, the sample was cooled to 600°C and then exposed to a watercontaining gas atmosphere (33% H₂O in argon). This leads to an oxidative mass increase of 7.4% with simultaneous release of hydrogen (mass number 2). Based on the abrupt changes in the course of the mass curve as well as the ion current curve of the mass spectrometer, it can be seen that the water splitting takes place as a multi-stage process and suggests a direct surface reaction as an initial reaction step followed by a diffusioncontrolled reaction in the second step.



Fig. 6. TGA-MS measurement plot illustrating the thermochemical water splitting process with a high-temperature oxide percursor (LSC20)

Conclusion

The platform concept of the NETZSCH STA 449 Jupiter[®] series together with the Aëolos[®] Quadro mass spectrometer offers an ideal basis for reproducing and studying complex thermally-induced reactions. In the abovementioned application example, a targeted investigation of thermochemical water splitting could be reproduced using the specially designed steam (or water-vapor) furnace and a steam generator. In addition to pure gravimetric detection, the processes taking place were also analyzed and interpreted with evolved gas analysis. Thus, the applied device combination provides the perfect foundation for a thermal characterization of the ongoing reactions in main current and future energy production routes.

In the final part of this energy-themed series (part 3, in our next **on**set edition), we will cover in detail the Pulse Thermal Analysis (*Puls*eTA®) technique for deconvoluting simultaneous chemical reactions not possible with conventional thermal analysis.

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TBB 913 – Fire Testing System for Floor Coverings

Dr. André Lindemann, Managing Director of NETZSCH TAURUS Instruments, Weimar



Fig. 1. TBB 913 fire testing system

Fire Protection in the Building Industry

Particularly in public facilities like airports, hospitals, child-care centers, schools and hotels, but also in shipping, aviation and in the private sector, it is extremely important to comply with fire-protection requirements. For the building materials industry, the fire behavior in terms of flammability, flame spread and smoke development of the materials employed is of particular importance.

Ideally, fires should be directly prevented or the spread of flames should be contained as well as possible, for example, by using appropriate floor coverings. In protecting life and limb to the greatest extent possible in the event of a fire, the time factor from ignition to the arrival of emergency services plays a decisive role.

Standards for Testing Fire Behavior

But how does one know whether the floor coverings employed meet fire-safety regulations? The DIN EN ISO 9239-1 standard, which is valid throughout Europe, stipulates mandatory tests for the fire behavior of floor coverings along with the classification of fire properties. With the new TBB 913 fire testing system for floor coverings by NETZSCH TAURUS Instruments, the fire behavior can be determined in order to classify floor coverings into the appropriate fire protection classes.

Functional Principle of the TBB 913

The TBB 913 is used to simulate typical fire scenarios, which can occur, for example, in long corridors in hotels and public facilities. A heat radiator in an inclined position generates the high thermal radiation energy on the flooring occurring in the event of a fire. The flooring specimen to be tested is laid out horizontally in the test chamber. The flame propagation distance, the smoke density as a function of time and the critical thermal radiation are determined. For tests that can be used for purposes of comparison and drawing conclusions, high reproducibility is required. The heat-flux profile needed for the specimen must be detected and precisely adhered to within narrow limitations in accordance with the standard.

For determination of the smoke gas density, transmission measurements (TRDA) along with temperature and pressure loss measurements are necessary to detect the gas volume flow. The gas velocity in the flue gas exhaust is also measured and constantly adjusted via speed-controlled fans. All types of floor coverings, such as carpets, wood, cork or plastic coverings, can be tested.



Fig. 2. Combustion chamber with tiltable radiator of the TBB 913

TBB 913

Classification in Fire Classes

Commercial and contract floor coverings employed in public or commercial buildings are tested in accordance with DIN EN ISO 9239-1 and EN 13501-1 (figure 3) for classification into the fire classes $A1_{\rm fl}$ to $F_{\rm fl}$.

Floor coverings are considered flame-retardant if they meet with the requirements associated with the radiation test for fire classes B_{fl} and C_{fl} . For products used in private homes, class E_{fl} is usually sufficient.

Intuitive Operation

The TBB 913 fire testing system operates via the "TBB Control" measurement and control unit. The integrated software serves for recording and processing the measured values and displaying the measurement results (figure 4). Measurement processes can be tracked and results can be displayed, saved, exported and logged in various formats (report generator).

Non-inflammable	A1 _{fl}
Non-initiatimable	A2 _{fl} - s1
Elamo rotardant	B _{fl} - s1
Hame-retaituant	C _{fl} - s1
	A2 _{fl} - s1
	B _{fl} - s2
Normally inflammable	C _{fl} - s2
Normally innaminable	D _{fl} - s1
	D _{fl} - s2
	Е _{fi}
Easily inflammable	F _{fl}

Fig 3. Fire-protection classes for floor coverings in accordance with DIN EN 13501-1*(s stands for smoke development)

*Source: https://www.cobaeurope.com/de/newsroom/ ratgeber-brandschutzklassen-fuer-bodenbelaege-nach-en-13501-12547



Fig. 4. Screenshot of the measurement course with the Windows-based TBB software

Further details can be found on our webiste at

https://analyzing-testing.netzsch.com/en/products/fire-testing/tbb-913

Determination of Oxygen Content in a Thermobalance

Dr. Jan Hanss, Head of Applications Laboratory

Factors Influencing the Oxygen Content

If the thermobalance is used under inert conditions, the oxygen content in your instrument seems to be negligible. However, even the residual content can have an influence on the results that is not insignificant. Therefore, the ASTM D6370 and ISO 9924 standards stipulate an O_2 content of less than 10 ppm. This raises questions such as: What does the O_2 content depend on and how can it be determined?

The O₂ content depends on the following factors:

- 1. Quality of the pressurized gas cylinder
- 2. Tightness of the thermobalance
- 3. Gas supply system to the thermobalance

Regarding 1: The nitrogen quality of 5.0 contains impurities of 10 ppm in the gas cylinder. Specification of the O_2 content is usually < 3 ppm. If an appropriate pressure reducer is additionally used and ensures proper cylinder replacement, the O_2 content is negligible.

Regarding 2: In many cases, there is no precise information on the oxygen uptake by means of the instrument.

Regarding 3: In buildings and/or laboratories with central gas supply, this consists of metal pipes to prevent O_2 diffusion. In contrast, in Teflon pipes with a diameter of 6 mm, approx. 10 ppm O_2 per running meter diffuse in at a gas flow of 100 ml/min.

How Can the Oxygen Content in a Thermobalance Be Determined?

If a gas analyzer is directly coupled to the outlet of a thermobalance, the O_2 content can be determined at this coupling point, for example, using an oxygen measuring probe by the company Zirox. This measures the O_2 content at a cell temperature of 750°C and is calibrated for a gas flow in the range of approx. 80 to 130 ml/min. With this approach, it should be noted that the occurrence of substances reacting with oxygen at the cell temperature, such as H_2 , CH_4 or other combustible gases, will lead to falsification of the measurement results.

Will any oxygen molecule in the gas flow come into contact with the sample? This is not to be expected, since the furnace cross-section is significantly larger than the crucible cross-section. This means for the user that, besides the O_2 concentration in the system, the proportion that reacts with the sample is also of interest.

How Can the Oxygen Content That Reacts with the Sample Be Determined?

Since experience has shown that activated carbons are not suited for determination, we recommend using a metal: in this case, a zirconium sheet (99.8%). This method was tested using a TG 209 *F1* Libra® with an SGM5 oxygen probe (Zirox) under an N₂ gas flow of 100 ml/min. To increase the O₂ content, the gas line to the instrument was expanded to approx. 4.3 m with the help of a Teflon hose.

The temperature dependence of the mass change can clearly be seen, although the O₂ concentration remains the same (upper curve, figure 1). The mass increase (with the example of a value at 1000°C) is solely due to the reaction with oxygen and amounts to 50 μ g at 1000°C in 30 min. This leads to an effective oxygen level of 12.7 ppm based on the following calculation:

$$n(O_2) = \frac{m(O_2)}{m(O_2)} = \frac{50 \,\mu g}{32 \,\frac{g}{mol}} = 1,56 \,\mu mol$$
$$V(O_2) = n(O_2) \cdot V_m(25^\circ C) = 1,56 \,\mu mol \cdot 24,4 \,\frac{l}{mol} = 38.13 \,\mu l$$

O₂ [Vol. ppm] = 38.13·10⁻³ml/(30 min·100ml/min)·1000000 = 12.7 ppm

Figure 1 additionally shows the zirconium measurement compared to O_2 values measured with the oxygen cell. The O_2 content in the empty measurement also changes with temperature due to such factors as impurities that only access the gas flow at higher temperatures or through varying levels of instrument tightness at different temperatures. To determine the O_2 consumption during the sample measurement with the oxygen measuring cell, the values produced by the empty measurement are subtracted at the individual temperatures.

Table 1 compares the measured O_2 content and the calculated O_2 consumption.

PRECISE PRACTICE

Table 1. Comparison of the measured and calculated concentration of oxygen

Temperature [°C]	Difference in O ₂ content of the measuring probe between: Empty – Zr sheet [ppm]	Mass increase Zr sheet [mg]	Calculated O ₂ consumption by Zr sheet [ppm]	Difference between the two O_2 determinations [ppm]
650	2	0.029	7.3	-5.3
750	9	0.034	8.6	0.4
850	8	0.035	8.9	-0.9
1000	13	0.050	12.7	0.3



Fig. 1. Measured O_2 concentration and the related mass change of the Zr sheet (green, at the top) as well as the course of the O_2 concentration of the empty measurement (black).

As can be seen, the measured values (oxygen measuring cell) and the calculated ones (Zr sheet) are in good agreement. Not the entire O_2 content in the carrier gas reacts with the sample. The contact between the carrier gas and the sample is additionally reduced by using a crucible with a lid (figure 2).

Using a crucible with a lid in thermogravimetric measurements generally affects the transfer of the gas both from the sample and to the sample.

Figure 3 depicts a comparison of measurements under common laboratory conditions, i.e., using a Teflon line, with the situation when using a steel line. Both experiments were carried out under an N_2 gas flow of 40 ml/min.

Summary

The O_2 content in a thermobalance can refer to either the entire O_2 content or just that reacting with the sample. If the reaction of this proportion with the



Fig. 2. Influence of a pierced lid and the conversion between the sample and oxygen in the carrier gas.



Fig. 3. Influence of a steel and Teflon line on the oxygen increase of zirconium.

sample is to be determined, a metal such as Zr is recommended. If it is a question of the general O_2 content in the instrument, an appropriate gas analyzer is the go-to solution.

If measurements show that the O_2 content in the thermobalance is too high, then even just improvements to the gas supply can have a positive influence on the result.

60 Years of NETZSCH-Gerätebau – 60 Years of Excellent Service

Andreas Strobel, Head of Global Customer Service



Fig. 1. Service by NETZSCH Analyzing & Testing: Your complete package from the provider

Excellence in Service

NETZSCH-Gerätebau GmbH has stood for customer orientation, professionalism, quality and sustainability for almost 60 years now. We want you to feel looked after in the best way possible – before and during the purchasing process, but also after sale. Our knowledgeable service team accompanies you throughout the entire life cycle of your analytical instrument to provide the most comprehensive support imaginable for problem-free long-term use.

Our service offerings (figure 1) will be there to help you quickly and efficiently at all times, especially in times of travel restrictions and advancing digitalization. Our vision is to provide you with the best possible backing at any given time and place.

Our service staff, both in the office and in the field, have been trained and educated in-house over many years.

Regular training ensures that our expert knowledge is always up to date. Accordingly, we offer our customers proficient technical service as well as a wide range of training options.

Thermal analysis instruments by NETZSCH-Gerätebau are also supported for an exceptionally long period of time. This was recently reflected in the results from the monthly raffles for "Who has the oldest instrument still in use?", which was a part of our 60th company anniversary celebration.

Prior to procurement, we clarify:

- What happens after the order?
- Who is my contact person in service?
- What are the next steps?
- What service options are there after the purchase?

60 Years of Excellent Service

Winning Dilatometer

The oldest dilatometer still in use (figure 2) is located at Wendel GmbH in Dillenburg, Germany. It was purchased in 1978 and has been reliably measuring the coefficient of ceramic frits, glazes and engobes ever since. Results from the last service check in November 2021: No defects!



Alongside other NETZSCH instruments, the oldest STA still in use (figure 3) is located at the Fraunhofer Institute for Ceramic Technologies and Systems, IKTS, in Dresden, Germany. This STA 429 was built in 1977 and has been used for investigations on debinding behavior and gas reactions in the production of ceramics and powder metallurgical materials, as well as the determination of transformation temperature and reaction enthalpies in processes such as melting and solidification.

By the way, information on our raffles and all articles about the "60 years of NETZSCH-Gerätebau" can be found on our blog:

https://ta-netzsch.com/



Fig. 2. Unrestricted use of the DIL 402 E/3 (1978) (Photo: ©Wendel GmbH)



Fig. 3. At the Fraunhofer IKTS in Dresden, an STA 429 from 1977 is still in operation and has been lovingly preserved and continuously updated over the years (Photo: ©IKTS Fraunhofer Dresden).

Customer Satisfaction Confirmed!

As recently as last year, the satisfaction of our customers was confirmed in a survey. Especially in terms of friendliness, reliability, speed and success rate, the results were convincing. With a vote-count of nearly 90%, the quality of our service and support offerings were deemed the second-most important criterion after product quality for customers' decisions to purchase a NETZSCH analyzer. We are grateful for this positive feedback!

In the Customer Service department, we also measure the quality of our service using various key figures. One of these key figures is the first-contemplation rate for service, which has been over 90% for years; in the last evaluation period, it was 93.8%. This means that almost all service calls were successfully concluded within the scope of the requirement.

We offer maintenance contracts that help control long-term costs, save money and preserve the value of your investment. We design training courses tailored to you – at your site, at our in-house training center or online via our NETZSCH Online Academy, **NOA.**

60 Years of Excellent Service

Tailor-Made Rheology Maintenance Contracts

We also offer customized solutions, for example, individualized application advice, assistance in certification and calibration service (figure 4).

Digital Service

In addition to classical digital service tools such as the hotline, chat and FAQ section on our website, NETZSCH offers a costeffective and efficient solution for your issue with the help of remote support. With the Team Viewer program (see figure 5), your technician can carry out minor adjustments and repairs to the instrument directly, while simultaneously receiving online technical support from one of our NETZSCH service engineers. This saves both time and costs.

1000 Years of Combined Experience

At NETZSCH, more than 100 service employees and representatives worldwide with over 1000 years of combined experience are on staff today to take care of you and your NETZSCH instruments. I have been responsible for worldwide customer service at NETZSCH-Gerätebau for more than 28 years, and would be very pleased to hear from you with any suggestions as to how we can tailor our range of services to even better meet your needs.

With us, you are always in the best hands, also in the next 60 years to come. Contact us any time!

Annual Maintence

Work and travel time including travel and accommodation costs is included Provision of a test certificate in accordance with ISO 11443 or DIN 53019 Use of monitored standards

Gold Maintenance Contract

Annual Maintenance plus

- Additional service visits, work and travel time are included
- Free software updates as part of the annual maintenance
- Provision of desktop and video remote service
- High priority for customer service and trouble shooting

Platinum Maintenance Contract

Gold Contract plus

- Additionally required spare and wear parts are included
- Optional provision of loaner equipment in the event of malfunction
- Highest priority for customer support and training

Fig. 4. Maintenance contracts for Kinexus and Rosand rheometers



Fig. 5. TeamViewer Assist AR, installed on a laptop and a smartphone

Mysterious Stories in the STA Customer Service in China

Ning Zhan, Vice President of NSI Service & Logistics

Ning Zhan has been Vice President of Service & Logistics at NETZSCH Scientific Instruments Trading (Shanghai) Ltd. (NSI) since 2012. He joined NETZSCH as a service engineer in 1998 and has seen the growth of NETZSCH Analyzing & Testing in China: From a service engineer to the head of 20 service employees in 2021 and from one office room in the German Center in Tongji University to the headquarters with more than 13 sales and service offices all around China.

Ning Zhan has already experienced a lot during his professional career at NETZSCH. We hope you will enjoy these funny stories about STA which have happened over the years in the NSI service team. Ning Zhan remembers three stories quite well:

German Quality



Approx. seven years ago, a user reported that the table in a lab collapsed and an STA 449 **F3** on it dropped down directly to the ground. "It's over!", the user thought, and asked our service to take a look and issue a "death certificate". Following a brief check and the exchange of the sample carrier, the STA returned to work immediately.

As you can see: German quality is not just a legend.

Shaking Instruments



Approximately four years ago, a user called for help that the TGA signal of the STA had jumped a little bit irregularly during several runs. The DSC curve, on the other hand, looked normal.

Our service tried to help at once: "Don't worry ... Please check the position of the sample carrier, the gas outlet, the chiller, the air conditioning above the device; is there any other big device working in the lab?" ... We spent all afternoon on it, but could not fix it. "OK, let's try tomorrow again."

The next morning, we were surprised when the user told us that the STA was now working normally. Neither of us could believe it. Later, we got the news that an earthquake of magnitude 5 had happened hundreds of kilometers away.

STA Friendship



In 2019, a colleague of mine visited a customer. "Happy birthday to you, ..." he heard people singing in a lab room. Oh, apparently some people are celebrating a birthday, but why celebrate in a lab? Is it allowed to have a party in a lab? Normally, a birthday party should be held in the break area or in a restaurant.

My colleague sneaked through the door: There was a big cake on a lab table in front of an instrument! Several persons were standing around it, but no person wore a birthday crown! The crown was on top of the furnace! What was going on here?

They were celebrating the birthday of a NETZSCH STA! The customer told us that this instrument was very important to them, as it helped a lot in their daily research work. Over the years, the instrument became one of their friends. Moreover, they wanted to treat their STA friend with care and affection.

So why not just celebrate its birthday! "Yes, if every instrument is being cared for properly, it will return more to you!"

Energy Solutions by NETZSCH – Battery Testing

Tailor-Made for Every Specific Product Requirement

Steven Min, President of NETZSCH Korea

The integration of renewable energy sources, such as wind and solar power, require efficient, reliable and flexible energy systems. Batteries are particularly suitable as energy storage systems. They are characterized by their unique ability to guickly absorb, store, and re-inject electricity. They are therefore a solution for maintaining and increasing system flexibility.

New challenges are on the horizon, and market needs are rapidly evolving, as are technologies and solutions for power protection, switching and conversion in energy storage systems.

Engineers face challenges when designing lithium-ion batteries. Proper thermal management strategy is required to ensure performance and a long life span of the battery. The main concern with the thermal behavior of room temperature batteries is the possible significant temperature increase that may cause thermal runaway.

Battery design parameters, manufacturing processes and operating conditions have an effect on the temperature rise/profile during battery operation.

Manufacturers of battery components must deliver consistent overall quality - throughout the entire manufacturing process. Quality and critical parameters that could affect battery performance should be monitored at every stage – from raw materials to cell assembly.

The development of powerful batteries with increased capacity, longer lifetime, shorter charging times and lower weight and size is becoming even more crucial due to the changes in mobility.

It is already apparent that market needs along with technologies and solutions for power generation and conversion in energy storage systems will continue to grow rapidly.

At NETZSCH, we are ready to support you with leadingedge technologies.

Various factors influence the manufacturing process and thus the battery performance:

- Chemical composition
- Shape and particle size distribution of the active materials

- Homogeneity and absence of defects in coatings on conductor foils of electrodes
- Transport and dosing of difficult aggressive media
- Feeding during mixing, grinding and dosing of cathode and anode slurries

The NETZSCH Group provides complete solutions for battery applications, from the grinding and dispersing of battery materials to stability, charging and discharging efficiency and even recycling.

Our equipment and instruments accompany both the overall manufacturing process and the research and development of Li-ion batteries.

Visit our new website, where you will also find our technologies for various areas of battery development as well as manufacturing:

www.energy.netzsch.com

PRODUCTION STEP Characterization of Components Accompanying the R&D Process





Grinding & Dispersing

ONset 24|2022 **25**

Batteries

Our Product Portfolio:

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- Dispersion
- Delamination
- Separation
- Deaeration
- Classification & spheroidization
- Pumping
- Thermal analysis & rheology
- Fire-testing systems



PRODUCTION STEP Raw Materials for Anode & Cathode

 \mathcal{T} CONDUCTIVITY LITHIUM PRECURSOR AGENT, BINDER, MIXING PROCESS SOURCE ADDITIVES 22 MIXING & WET GRINDING PROCESS COAT, DRY, CALENDER \mathcal{T} \approx HEAT TREATMENT, CHEMICAL PRO-CESSING POST PROCESS WET GRINDING & DRY GRINDING & DEAGGLOMERATION DEAGGLOMERA-TION INCL. CLASSIFICATION \mathcal{T} MATERIAL CHARACTERIZATION DRYING \mathcal{N} λ BATTERY ACTIVE MATERIAL

PRODUCTION STEP Conductive Additives & Battery Slurry

Outlook

Coming Soon ...

We will soon be releasing our new applications booklet 'Thermal Properties of Metals and Alloys'!

This new booklet presents an introduction into the broad applications spectrum of thermoanalytical methods and illustrates how these can be used in determining thermophysical properties of metals and alloys.

After a brief outline of measurement equipment and methods, practical measurement examples on metals and alloys are shown and the effects interpreted. The work is intended to serve as both an introduction and an aid to practitioners and researchers alike.

Don't hesitate to contact us and reserve your copy!

www.netzsch.com/handbook-metal-and-alloys



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Simply register for any of our webinars on our homepage free of charge. Below is a small selection for June. You can also find additional webinars on our homepage at:

www.netzsch.com/webinars



June 2, 2022: DSC Performance at High Temperatures 10:00 a.m. in German (CET time zone) 4:00 p.m. in English (CET time zone)

June 7, 2022: Live from the Lab – High-Temperature Ceramics 8:00 p.m. in English (CET time zone)

June 21, 2022:

Carbon Content Analysis of LiFePO₄ Cathode Material 9:00 a.m. in English (CET time zone)

June 29, 2022: Evolved Gas Analysis by Mass Spectrometry – How Mass Spectrometry Can Be Used in Combination with Thermogravimetry 9:00 a.m. in German (CET time zone) 4:00 p.m. in English (CET time zone)



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Imprint

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