

Differential Scanning Calorimetry – DSC

Method, Technique, Applications



DSC 200 **F3** *Maia*[®] – Principle of Operation

Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) is one of the most frequently used techniques in the field of thermal characterization of solids and liquids.

The DSC method can be used for the analysis of energetic effects such as:

- Melting/crystallization behavior
- Solid-solid transitions
- Polymorphism
- Degree of crystallinity
- Glass transitions
- Cross-linking reactions
- Oxidative stability
- Decomposition behavior
- Purity determination
- Specific heat

In this technique, a sample is placed inside a crucible which is then placed inside the measurement cell (furnace) of the DSC system along with a reference pan which is normally empty. By applying a controlled temperature program (isothermal, heating or cooling at constant rates), caloric changes can be characterized.

Easy handling and rapid analysis are among the hallmarks of this analytical technique, which has proved to be highly significant in the areas of research, development, and quality control. There are a variety of standards (ASTM, DIN, ISO, etc.) for the application, evaluation and

interpretation of specific materials, products and properties.

The DSC 200 **F3** *Maia*[®] combines the advantages of modern technology, high sensitivity and a robust, easy-to-operate work horse. Tests can be carried out in the maximum temperature range between -170°C and 600°C.

The key components of the DSC 200 **F3** *Maia*[®] are the DSC heat flux sensor and a special furnace.

The sensor of the DSC 200 **F3** *Maia*[®] combines high stability, improved resolution and fast response time. Laser-guided welding processes for the sensor disks and thermocouple wires yield true sensitivity and robustness.

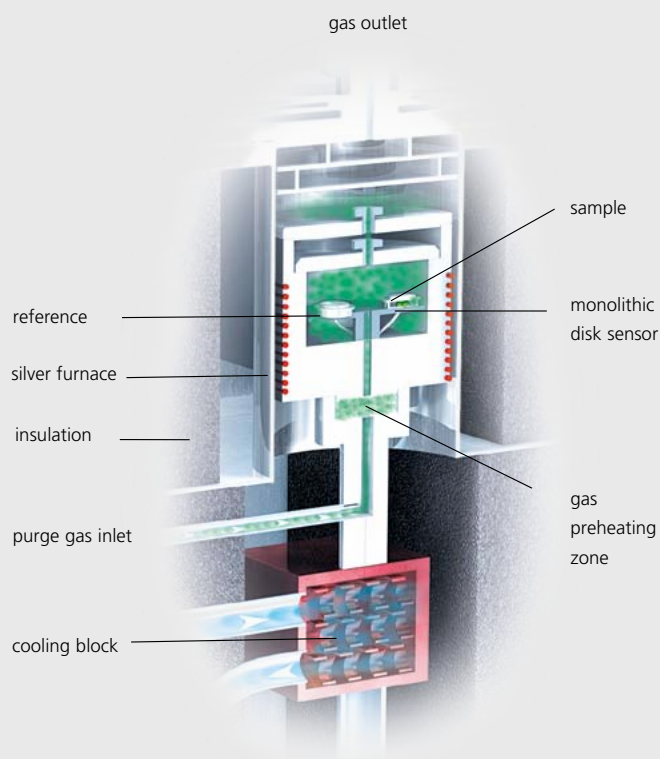


DSC 200 **F3** *Maia*[®] with Automatic Sample Changer (ASC)

The heating wires of the furnace surround the entire sensor plate. They are arranged in such a way that no temperature gradients occur in or above the sensor disk. This arrangement is the basis for a highly homogeneous heat flow to the sample and reference pans from all sides and therefore also for a highly stable baseline and an excellent signal-to-noise ratio. Protective and purge gas inlets are, of course, standard features of the unit.

For improved cooling times and subambient temperature tests, various cooling options such as forced air, intracooler or liquid nitrogen cooling systems are available. Of course, a versatile gas switching and flow control system are also available.

For routine applications we offer an Automatic Sample Changer (ASC) for up to 20 samples and references, which accommodates different crucible types.



Technical Specifications

Temperature range	-170°C to 600°C
Heating rates	0.001 K/min to 100 K/min
Cooling rates	0.001 K/min to 100 K/min (depending on temp.)
Sensor	Heat flux system
Measurement range	0 mW to ±600 mW
Temperature accuracy	0.1 K
Enthalpy accuracy	< 1%
Cooling options	Forced air, Vortex tube (up to 0°C) LN ₂ (up to -170°C) Intracooler -40°C or -70°C
Atmospheres	oxid., inert (static, dynamic)
Automatic Sample Changer	up to 20 samples and references (optional)

Additional Information

www.netzsch.com/dsc200f3

Proteus® Software for the DSC 200 **F3 Maia**®

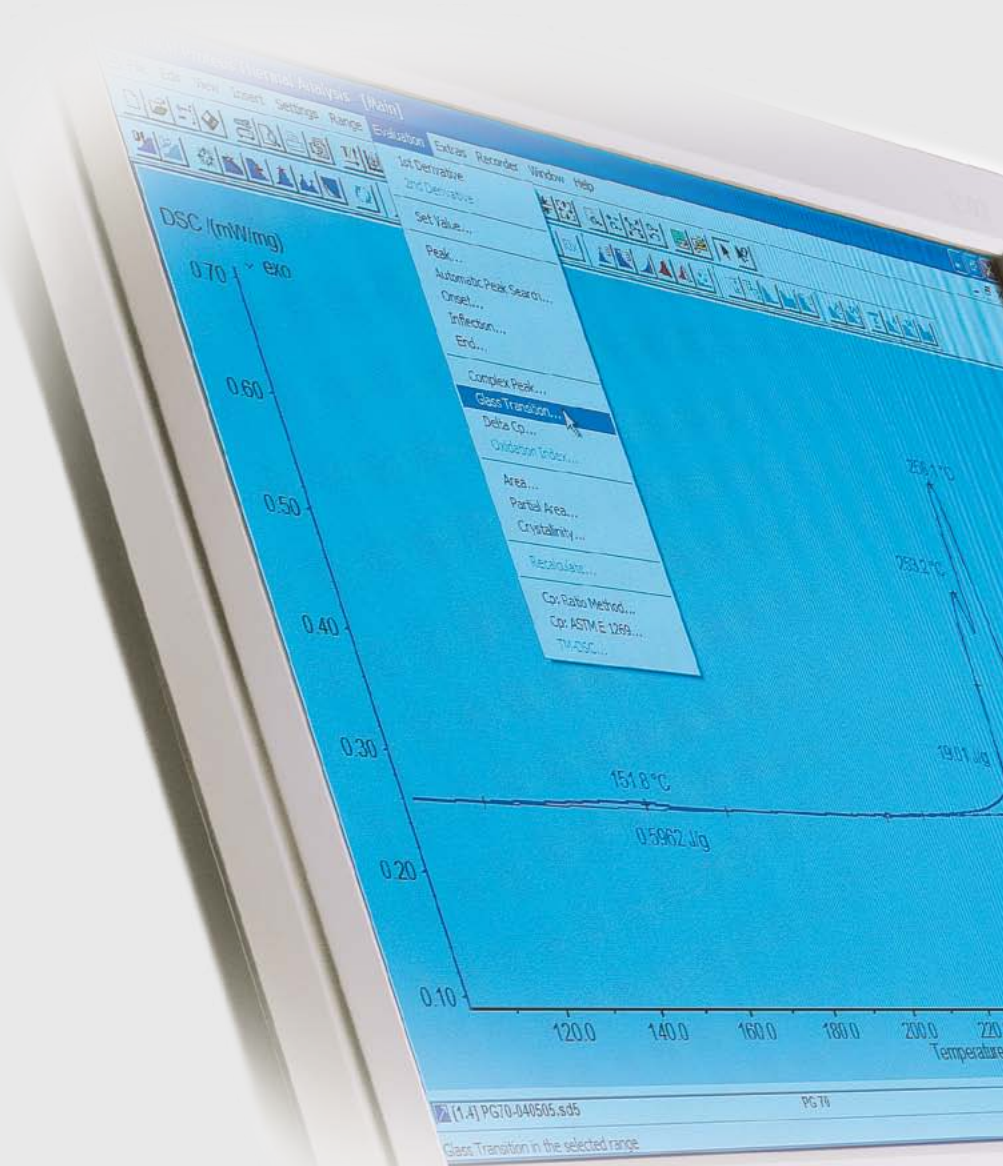
The DSC 200 **F3 Maia**® runs under a Windows® operating system which includes everything you need to carry

out a measurement and evaluate the resulting data. User-friendly menus combined with automated routines

make *Proteus*® very easy to use while still providing sophisticated analysis.

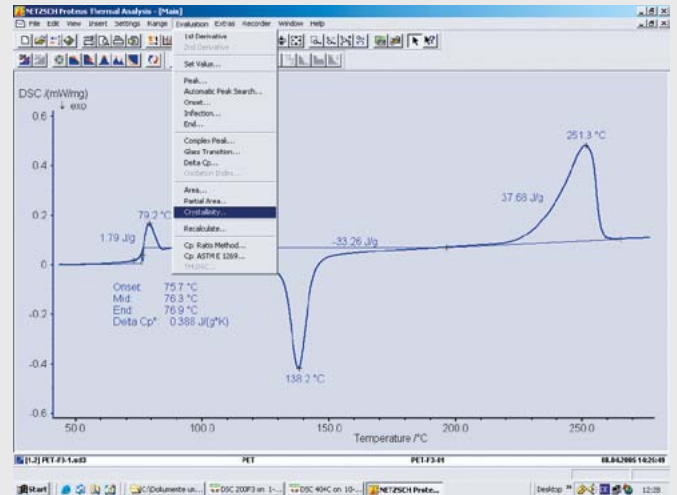
General Software Features

- Multi-tasking: simultaneous measurement and evaluation
- Multi-moduling: operation of different instruments with one computer
- Combined analysis: comparison and/or evaluation of DSC, TG and TMA and DMA measurements in one plot
- Labeling: input and free placement of text elements
- Calculation of 1st and 2nd derivative
- Selectable scaling
- Graphic and data export
- Selectable colors and line types
- Storage and restoration of analyses
- Macro recorder (optional)
- Context-sensitive help system
- Temperature calibration
- Compatible with advanced software packages (*Peak Separation, Thermokinetics*)
- Software produced by ISO-certified company



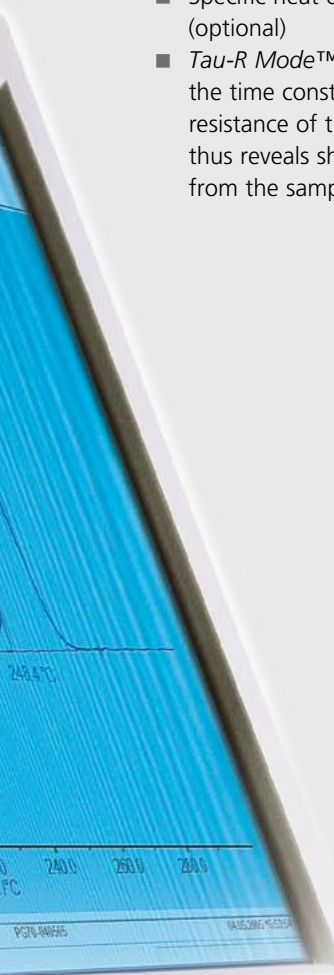
DSC Features

- Determination of onset, peak, inflection and end temperatures
- Automatic peak search
- Transformation enthalpies: analysis of peak areas (enthalpies) with selectable baseline and partial peak area analysis and consideration of mass changes
- Comprehensive glass transition analysis
- Automatic baseline correction
- Degree of crystallinity
- Specific heat determination (optional)
- *Tau-R Mode*TM: takes into account the time constant and thermal resistance of the instrument and thus reveals sharper DSC effects from the sample



Advanced Software (optional)

- *Peak Separation* for accurate separation and evaluation of overlapping transitions
- *NETZSCH Thermokinetics* for advanced characterization of reactions and kinetic parameters on the basis of multiple-step kinetic analysis on up to 16 curves; also provides predictions of the process



DSC 200 **F3** *Maia*[®] – Applications

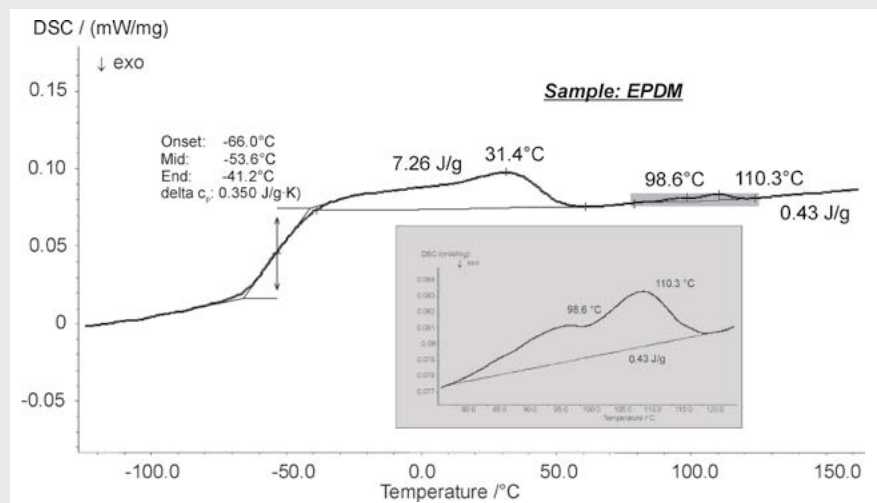
The DSC 200 **F3** *Maia*[®] can be employed for the characterization of a great variety of materials and applications including polymers, pharmaceuticals, textiles, foods, cosmetics, and the like.

For researchers in fields such as automotive, clothing, drugs, and so on, the technique employed by this instrument is a fast and reliable research tool. Furthermore, due to the easy operation,

fast analysis time and standardized evaluation procedures, the DSC 200 **F3** *Maia*[®] is optimized for application in quality assurance and failure analysis laboratories.

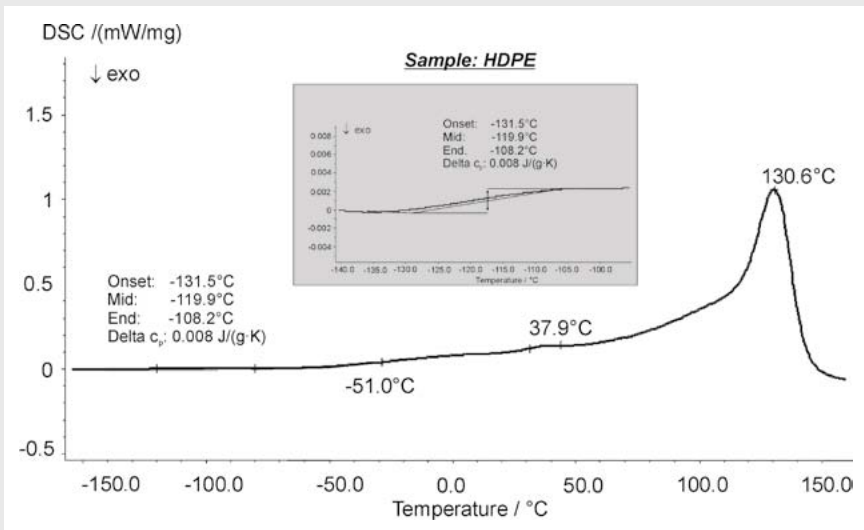
Ethylene-Propylene-Diene-Rubber-Mixture (EPDM)

The thermal behavior of an EPDM rubber mixture was measured between -125°C and 160°C at 10 K/min. The glass transition was detected at -53.6°C. The melting above the glass transition (peak temperature at 31.4°C) is typical for the behavior of a sequence-type EPDM. The further endothermic effects (at 98.6°C and 110.3°C) are due to the evaporation of processing agents. The zoomed graph in the inset clearly shows the high sensitivity of the DSC 200 **F3** *Maia*[®] even for small energetic effects (0.43 J/g).



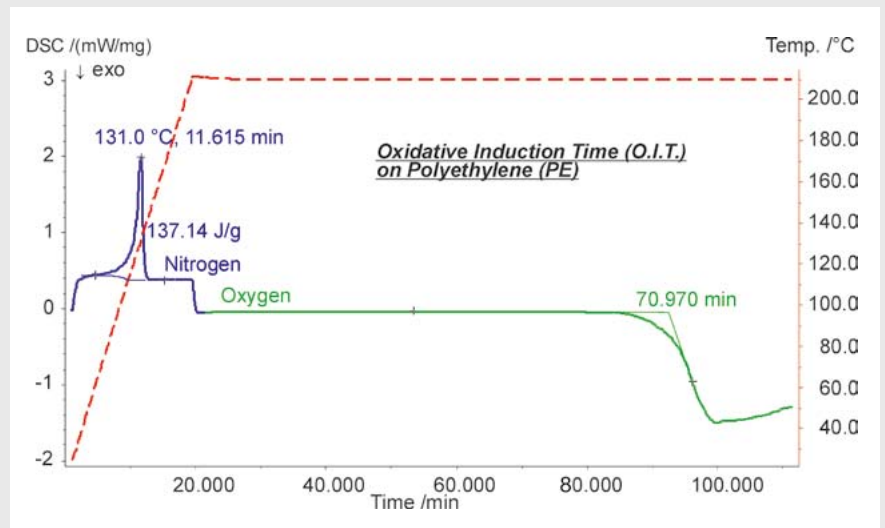
Polyethylene (PE)

PE materials such as high-density polyethylene (HDPE) are often used for the production of containers for packaging. Differential scanning calorimetry is often used to characterize the melting behavior of such materials, but the DSC 200 **F3 Maia**® can do even more. Due to its excellent low-temperature performance and outstanding sensitivity, the system also allows the detection of the glass transition (at -119.9°C). This extremely weak step in the DSC curve is presented in the inset in more detail.



Oxidative Induction Time (O.I.T.)

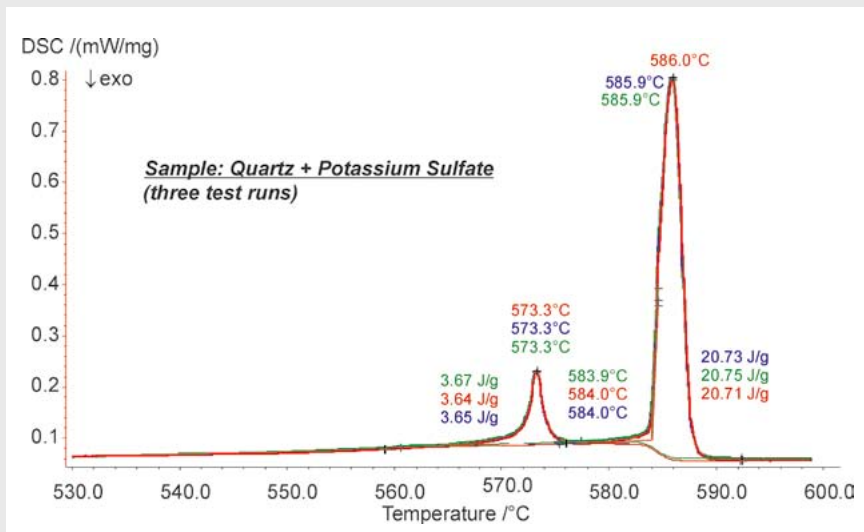
Analysis of the thermal stability of polymers is important for materials under thermal load such as polymer insulation for electronics. Determination of the oxidative induction time (O.I.T.) is a standardized technique which can be easily carried out using the DSC 200 **F3 Maia**®. Presented here is a measurement on PE heated to 210°C under inert (nitrogen) conditions. After a five-minute equilibration time, the atmosphere was switched to oxygen; the sample then began to degrade after 71 minutes under oxidizing conditions.



DSC 200 **F3** Maia[®] – Applications

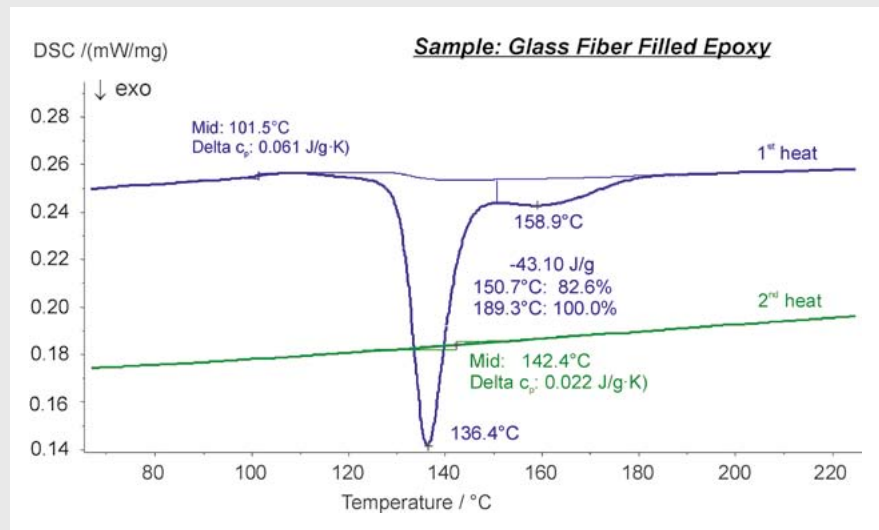
High-Temperature Applications

The DSC 200 **F3** Maia[®] allows tests up to 600°C with excellent baseline stability and reproducibility. Presented here are three runs on a mixture of crystalline SiO₂ (quartz) and potassium sulphate (K₂SO₄). The result clearly proves the outstanding temperature and enthalpy reproducibility of this cost-effective differential scanning calorimeter even at the limits of the temperature range.



Curing of a Thermosetting Resin

Analysis and optimization of the curing process of thermosets can be easily carried out using differential scanning calorimetry. Presented here is a measurement on a glass fiber filled epoxy measured in the DSC 200 **F3** Maia[®]. The two-step exothermic cross-linking reaction slightly above the glass transition (at 101.5°C) is clearly visible during the first heating of the sample. After a controlled cooling at 5 K/min, the sample was heated a second time. In contrast with the first heating, the glass transition is shifted to 142.4°C in the second heating.



DSC Accessories

The DSC 200 **F3 Maia**® can be equipped with various accessories and add-ons for optimum adjustment of the system to your requirements. Various cooling systems can be used to cool the furnace back to room temperature. Subambient temperatures (down to -70°C) can be achieved with the cost-effective

Intracooler. The liquid nitrogen cooling system allows tests at subambient temperatures down to -170°C. The DSC 200 **F3 Maia**® can be equipped with a gas flow control system for precise control of up to three different purge/protective gases. Routine measurements are facilitated with the

convenient automatic sample changer (ASC) for up to 20 samples and references, even in different crucible types. A wide range of crucibles (aluminum, silver, gold, copper, platinum, alumina, zirconia, graphite, stainless steel, etc.) is available for nearly all possible applications and materials.



Sealing press for different aluminum crucible types

Calibration kit

Crucible variety

Expertise in Service



Our Expertise – Service

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, before and after sale. Our qualified personnel from the applications, technical service and training 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.

To maintain and protect your investment, you will be accompanied by our experienced service team over the entire life span of your instrument.

Summary of Our Services

- Installation and commissioning
- Hotline service
- Preventive maintenance
- Calibration service
- IQ /OQ/PQ
- On-site repairs with emergency service for NETZSCH components
- Moving/exchange service
- Technical information service
- Spare parts assistance

Our Expertise – Applications Laboratories

The NETZSCH Analyzing & Testing applications laboratories are proficient partners for nearly any Thermal Analysis issue. Our diverse measuring methods and over 30 different state-of-the-art test stations will provide ready solutions for all your thermal needs.

Measurements can be carried out on samples of the most varied of geometries and configurations. You will receive high-precision measurement results and valuable interpretations from our expert team. This will enable you to precisely define new materials and components before actual deployment, minimize risks of failure, and gain decisive advantages over your competitors.

For production problems, we can work with you to analyze causal issues and work out solution concepts. The option of commercial testing is a relatively low investment which generally pays itself off in a few days or weeks and provides fast and reliable support for research on new materials or resolving any kinds of production issues.



The NETZSCH Group is an owner-managed, internationally operating technology company headquartered in Germany.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 2,500 employees at 130 sales and production centers in 23 countries across the globe guarantee that expert service is never far from our customers.

When it comes to Thermal Analysis, Adiabatic Reaction Calorimetry and the determination of Thermophysical Properties, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42
95100 Selb
Germany
Tel.: +49 9287 881-0
Fax: +49 9287 881-505
at@netsch.com

www.netsch.com