NETZSCH
Proven Excellence.

Instruments for Testing Refractories

Refractories under Load and Creep in Compression – RUL/CIC 421

Analyzing & Testing

REFRACTORIES

Today, selection of the proper refractory material is more crucial than ever when it comes to improving the cost-effectiveness of a process and prolonging the life cycle. Appropriate selection of the refractory furnace lining can only be made with accurate knowledge of the properties of the refractory materials and the stresses on the materials during service.

Thermomechanical properties are determined using high-temperature test methods with external forces causing stresses on the tested material. The stress-strain behavior of refractories at high temperatures includes reversible elastic strain as well as non-reversible time-dependent deformations. Therefore, the thermomechanical behavior of refractories must be considered as an interaction of stress, strain, temperature and time.

The testing of refractories – clearly an essential process – includes the following applications:

- Selection of material
- Characterization of new materials
- Prediction of service conditions
- Quality control of the process and the product
- Failure analysis
- Mathematical modeling for product improvements

The efficiency of any technical furnace plant depends – for the most part – on the quality of the refractory material and the correct installation of the furnace lining.

NETZSCH Instruments for Testing Refractories under Thermal and Thermomechanical Stress

Thermal and Thermomechanical Properties	Instrument	Temperature Range
Refractoriness under Load (RUL) Creep in Compression (CIC)	Refractoriness under Load/ Creep in Compression (RUL/CIC 421)	RT to 1700°C
Thermal Expansion (DIL) Volume Stability (DIL)	Dilatometer (DIL 402 E, DIL *Expedis* series)	-180°C to 2800°C

Thermal Stress	Instrument	Temperature Range
Specific Heat Capacity (LFA)	Laser/Light Flash Apparatus (LFA 427, LFA 457 *MicroFlash*®, LFA 467 *HyperFlash*®)	-125°C to 2800°C
Specific Heat Capacity (DSC)	Differential Scanning Calorimeter (DSC 404 ***F1/F3*** *Pegasus*®)	-150°C to 1400°C (2000°C)
Thermal Diffusivity/ Thermal Conductivity (LFA)	Laser/Light Flash Apparatus (LFA 427, LFA 457 *MicroFlash*®, LFA 467 *HyperFlash*®)	-125°C to 2800°C

International Standards for Testing Refractory Materials

Standard	ISO	DIN EN	ASTM	Instrument
Determination of Refractoriness under Load (Differential—with Rising Temperature)	1893	993-8		RUL 421
Determination of Creep in Compression (CIC)	3187, 16835	993-9	C832-00	RUL 421

RUL/CIC 421

Refractoriness Under Load and

RUL/CIC 421

Refractoriness under load evaluates the behavior of fired refractory bricks under rising temperature and constant load conditions.

Method

Refractoriness under load (RUL) is a measure of the resistance of a refractory product to subsidence when subjected to the combined effects of load, rising temperature at a predefined heating rate. The range in which softening occurs is not identical with the melting range of pure raw materials, but it is influenced by the content and the degree of distribution of low melting point fluxing agents.

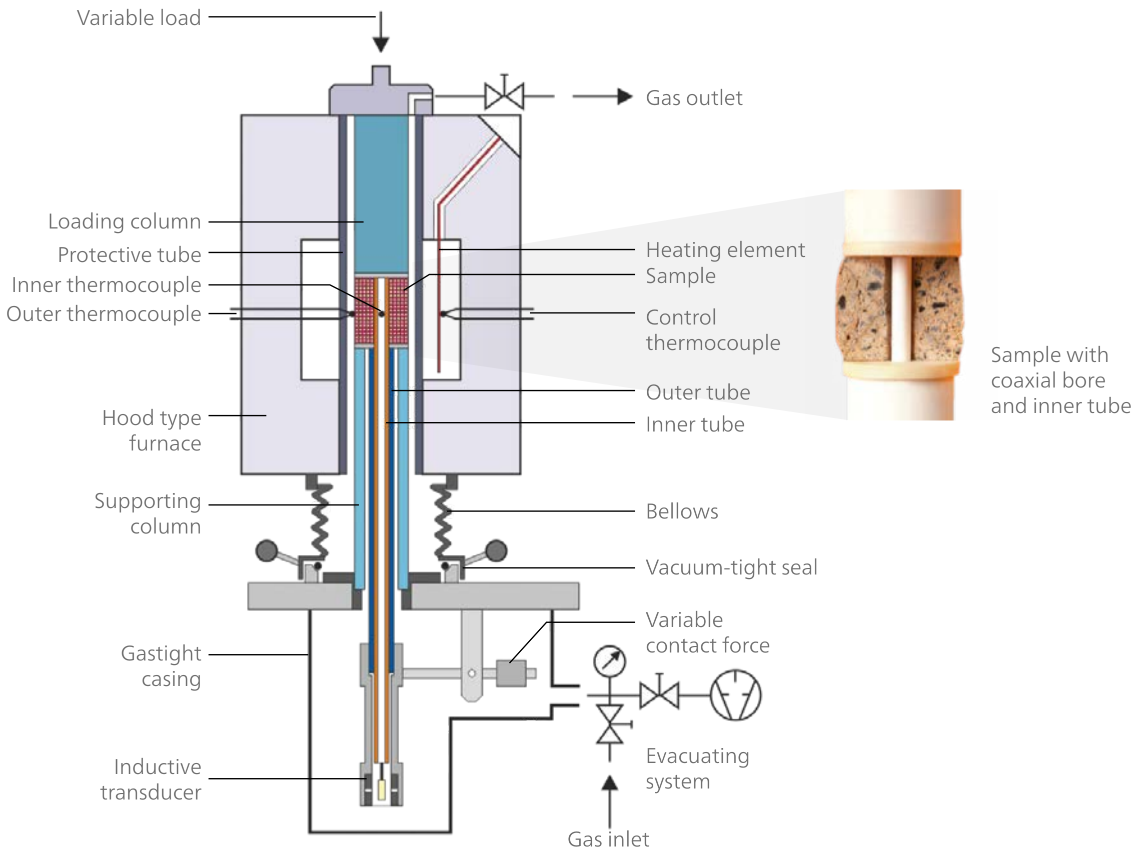
RUL

The RUL test method is described in ISO 1893, Refractoriness under load (RUL, differential – with rising temperature). A cylindrical test piece (50 mm in Ø and height with coaxial bore of 12.5 mm) is subjected to a specified constant compressive load and heated at a specified rate until a prescribed deformation or subsidence occurs. The deformation of the test piece is recorded as the temperature increases, and the temperatures corresponding to specified proportional degrees of deformation are determined.

CIC

The instrument can also be used for the determination of creep in compression (CIC) as described in ISO 3187. A cylindrical test piece is heated under specified conditions (see RUL) to a given temperature. While being held at that constant temperature, the deformation of the test piece is recorded and the percentage change is evaluated as a function of time.

Creep in Compression



Schematic of the gas-tight RUL/CIC 421 for measurements in protective gas atmosphere

Measuring Unit – Specimen

The measuring unit consists of a console, furnace guide frame, furnace (max. 1700°C), balance weight of the loading device (max. 1000 N) and a differential measuring system. To prevent chimney effects and to guarantee a sufficient zone of constant temperature, the furnace is closed at the top. The test piece is placed on the supporting column; the furnace is lowered and load is applied by counterbalancing (minimum of 100 cN increments) the weight of the furnace. Thermocouples serve to control the furnace and to determine the test piece temperature.

Signal Generation – Expansion

The length change is transmitted by a measuring system consisting of an inner and outer tube which act differentially. These tubes made of alumina are connected to an inductive transducer. Its signal is amplified and stored after A/D conversion. The thermal expansion of the tubes is self-compensating. Therefore, only the expansion of that part of the inner tube which corresponds to the test pieces must be considered. The differential measuring system can be used to measure the length change of the test piece through its center bore (ISO/DIN) or along the outside.

RUL/CIC 421

Gas-tight version for testing oxygen-sensitive specimens

Test Atmosphere

Measurements can be carried out in static air (basic version) or using an optional device for inert gas purge within the test piece area.

Testing Carbon-Containing Materials

For testing carbon-containing materials (e.g., magnesia-carbon graphite bricks), a non-oxidizing test atmosphere can be realized with a gas-tight test chamber (optional; see figure on previous page). This chamber can be evacuated and then purged with protective gas. Measurements can be carried out up to 1600°C.

Variable Loading Device

Optionally, the loading device can be equipped for load variation. The preload can be up to 300 N and the verifying load can be applied in the range from 0 N to 700 N at a rate between 0.3 N/s and 3 N/s.

Test Piece Dimension

Generally, identical test piece dimensions of 50 mm in diameter and 50 mm in height are used for both the RUL and the CIC test. For the high-precision differential measuring system used in determining expansion and deformation, the cylindrical test piece has a coaxial bore of 12.5 mm. The ground faces should be plane, parallel and perpendicular to the axis of the cylinder (ISO/DIN). Other test piece dimensions are also possible (e.g., 36 mm, GOST 4070-20000). .

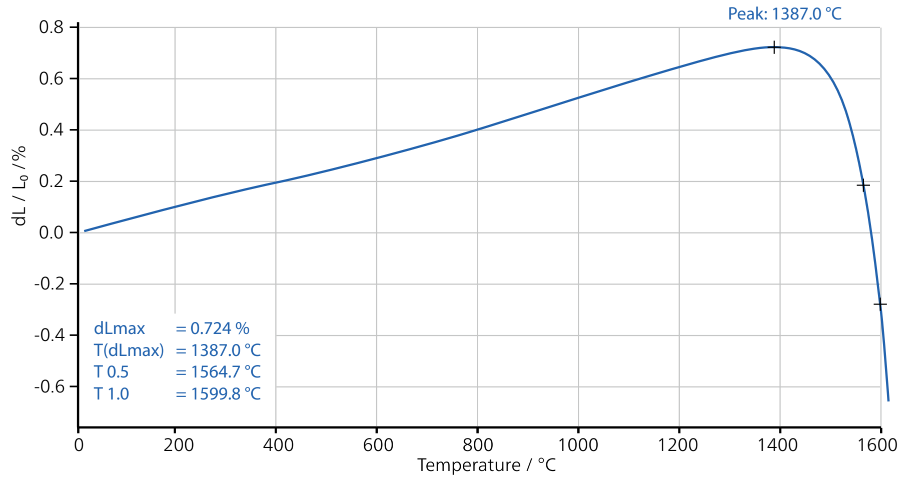
Sample Preparation Machines

NETZSCH offers the following machines for the appropriate preparation of optimum test pieces:

- Drilling machine 421/11
- Grinding machine 421/12
- Sawing machine 421/13

Evaluation Routines for RUL and CIC

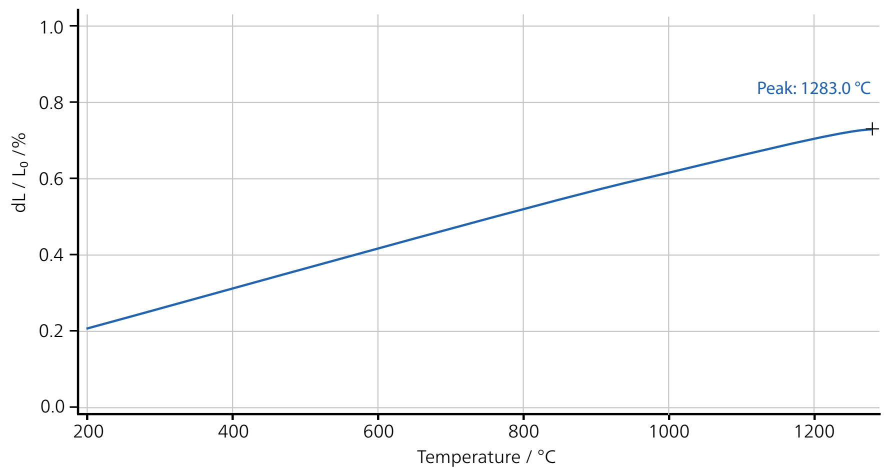
The upper plot shows a RUL measurement (differential) on a test piece of a fireclay brick with increasing temperature. At 1387°C, the test piece reaches its maximum expansion. Deformations of 0.5% and 1.0% occurred at 1565°C (T0.5) and 1600°C (T1), respectively.



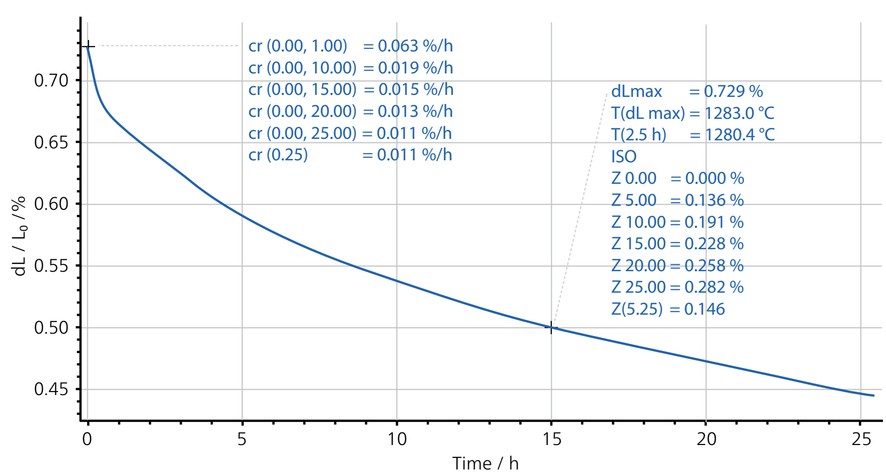
Refractoriness under Load; test conditions: 0.2 N/mm², 5 K/min, static air

The lower two plots depict a CIC measurement on a test piece of a silica brick. In the upper plot, the dynamic heating segment is presented.

The lower one shows the time-scaled creep over 25 hours at a constant temperature of 1280°C.



Creep in Compression; heating segment at a heating rate of 5 K/min



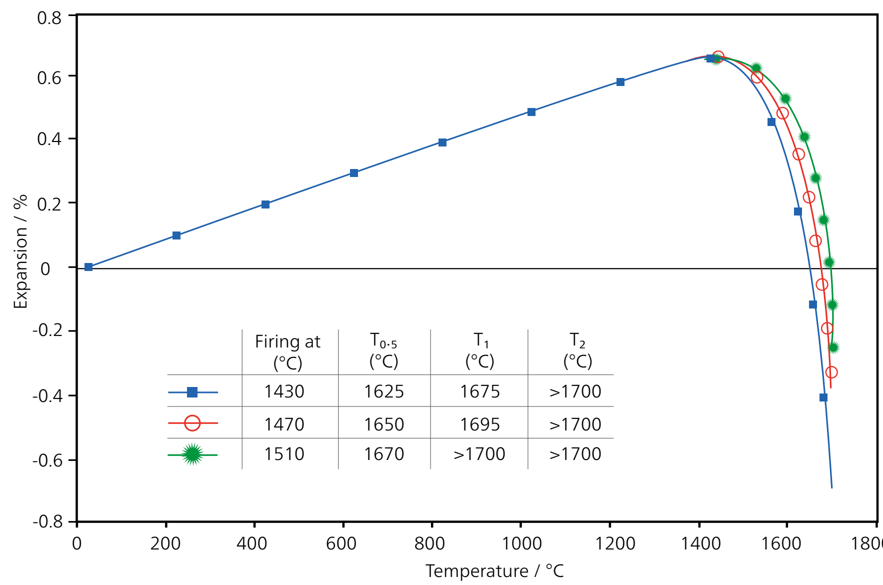
Creep in Compression; isothermal creep at 1280°C, 25 h in static air

RUL/CIC 421

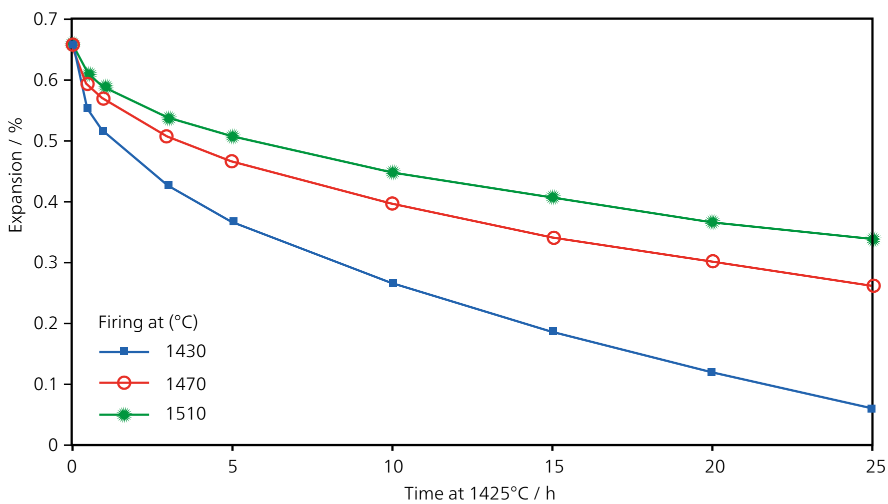
Applications

Refractoriness under Load (RUL) of Andalusite Bricks

This plot shows three measurements on andalusite bricks fired at three different temperatures: 1430°C, 1470°C and 1510°C. At approx. 1425°C, all three test pieces reach their maximum expansion. After applying sample or calibration curve correction, the software calculates the characteristic $T_{0.5}$, T_1 and T_2 temperatures from the RUL tests where 0.5%, 1% or 2% shrinkage is reached after the maximum expansion. The influence of the firing temperature can be clearly seen.



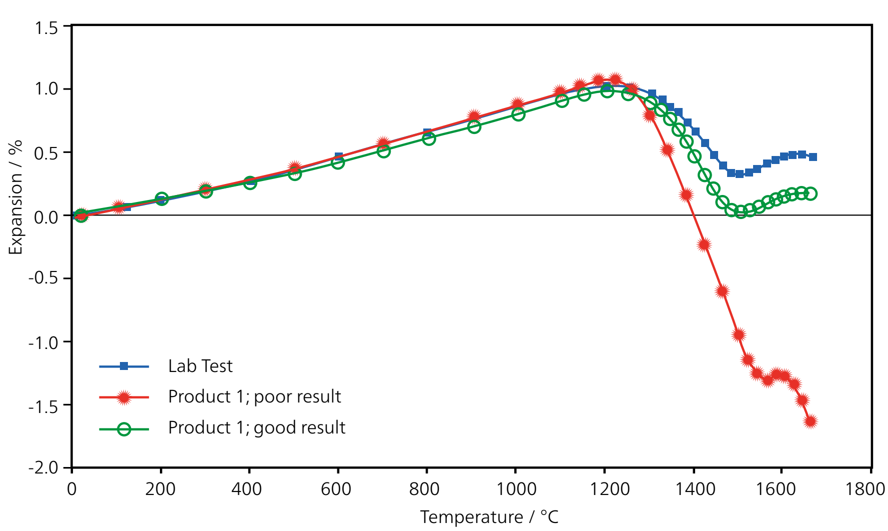
RUL behavior of andalusite bricks (approx. 65% Al_2O_3) fired at three different temperatures; test conditions: 0.2 N/mm², 5 K/min, static air



CIC behavior of andalusite bricks (approx. 65% Al_2O_3) fired at three different temperatures; test conditions: 0.2 N/mm², 5 K/min, static air, 25 h at 1425°C

Creep in Compression (CIC) of Andalusite Bricks

Test pieces of the andalusite bricks fired at different temperatures are used for CIC tests. In these tests, the load is applied once the temperature is reached (here, at 1425°C). This is contrary to RUL tests, where the load is applied from the very beginning of the measurement. This plot only shows the time-scaled creep at constant temperature (the heating segment is not depicted).

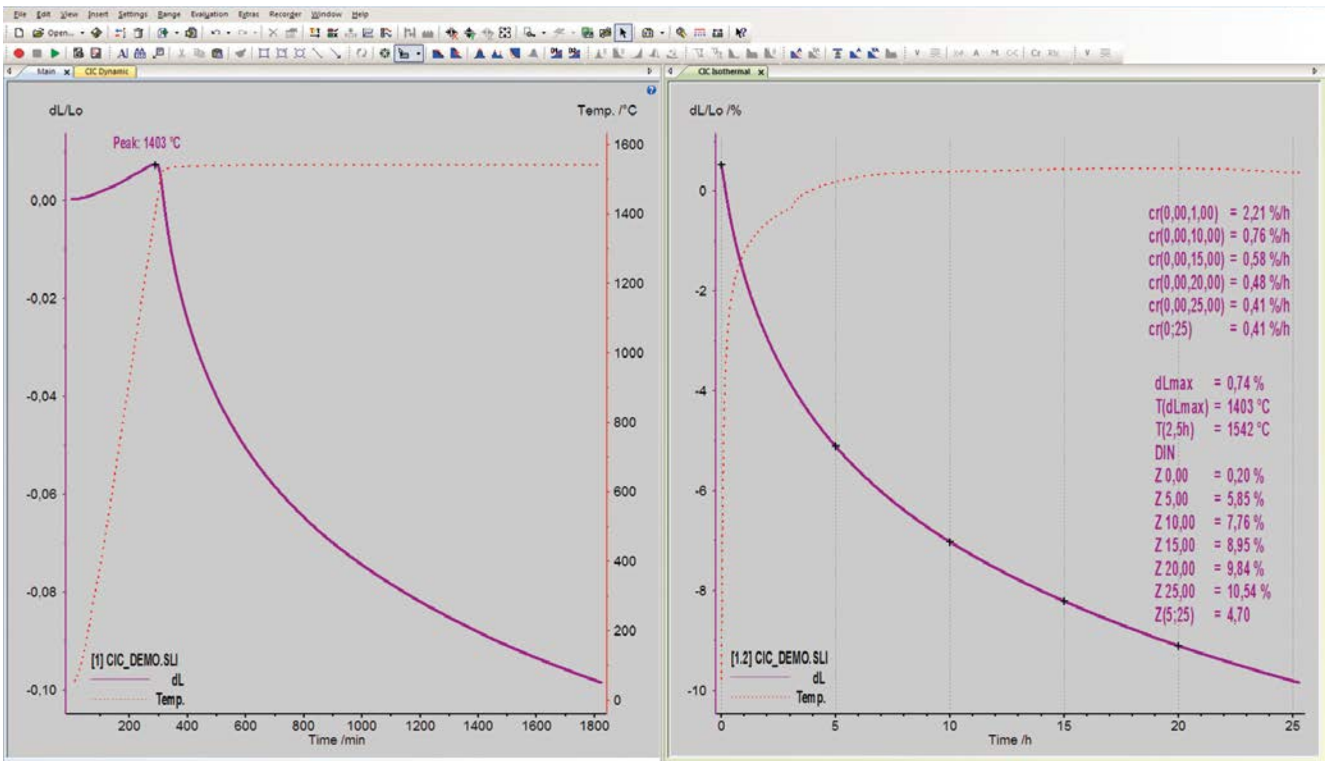


RUL test as quality control for a finished construction part

Quality Control of Castable Refractories by RUL Tests

Castables are mixed with water and then installed by either pouring or pumping. These measurements were carried out on finished construction parts. Castables have to be prepared exactly as instructed by the product manufacturer. Field tests show that the product depicted here by the red sample curve was improperly handled. The divergent shrinkage behavior (as compared to the blue and green curves) causes failures in the finished product.

RUL/CIC 421
Software



State-of-the-Art Software Including Various Evaluation Routines

- Graphic and tabulated results, calculated according to ISO/DIN
- Correction of the measured data by calibration curves
- Determination of characteristic data according to user's requirements
- Automatic softening point detection
- Derivation of curves for determination of the temperature- or time-dependent linear expansion rates
- Possibilities for temperature control (max. 96 isothermal or dynamic temperature program steps)
- Presentation of the measuring values temperature- or time-scaled for RUL and time-scaled for CIC
- Determination of the absolute maximum in the length dilatation spectrum (RUL)
- Determination of the sample temperature 2.5 h after start of isothermal phase (CIC)
- Determination of relative length changes at preset times
- Calculation of creep rates in preset time intervals
- Simultaneous analysis of up to 8 curves/temperature segments (curve comparison)
- Calculation of single values of physical or technical expansion coefficients
- Calculation and graphic display of the 1st and/or 2nd derivative, peak determination

Technical Specifications

The RUL/CIC runs under *Proteus*® software on Windows® for the fully automatic test run, data acquisition, storage and offline evaluation.

- Semi-automatic routines for the determination of reaction steps as extrapolated onset, point of inflection, peak, peak end
- Output or ASCII file export of the corrected measuring data
- Graphic export
- Data transfer of the sample length from an external gauge (optional)

	RUL/CIC 421 E/6
Temperature range	RT to 1700°C
Heating elements	4 Super-Kanthal 1800
Test atmosphere	Static air; optional inert purge gas
Safety switch	Failure of test piece
Test piece	Ø 50 mm, height 50 mm
Load range	1 N to 1000 N; steps of 1 N to 100 N
Max. stress	0.5 N/mm^2
Measuring range	20 mm; resolution 4,000,000 steps
Measuring system	Differential
Digital resolution	5 nm
Thermocouples	Type B
Power Supply Electronics	▪ Electronics : 230 V/10 A 50 Hz ▪ Furnace: 230 V/70 A/50 Hz; max. 15 kW
Dimensions	▪ Measuring unit: ≈ 1200 mm x 610 mm x 2400 mm ▪ Control unit: 562 mm x 555 mm x 1183 mm
Weights	▪ Measuring unit: ≈ 480 kg ▪ Control unit: ≈ 220 kg

The owner-managed NETZSCH Group is a leading global technology company specializing in mechanical, plant and instrument engineering.

Under the management of Erich NETZSCH B.V. & Co. Holding KG, the company consists of the three business units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems, which are geared towards specific industries and products. A worldwide sales and service network has guaranteed customer proximity and competent service since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 60 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.

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