GNR OPTICA

ANALYTICAL INSTRUMENTS GROUP

# EUROPE THETA/THETA


## BENCHTOP X-RAY DIFFRACTOMETER

www.gnr.it

# About us

**G.N.R. S.r.l.**, thanks to its 30 years of experience, is a worldwide market manufacturer of advanced analytical instruments, developing procedures of analysis for various applications, supplying the corresponding laboratory equipment and providing consulting and Customer support worldwide, through its well established sales and post-sale network.

**G.N.R. S.r.l.** projects and manufactures Optical Emission Spectrometers (OES) and Rotating Disc Electrode Optical Emission Spectrometers (RDE-OES) for the measurement of elemental composition of metal alloys and the analysis of contaminants, additives and wear metals in oils and lubricants, coolants and hydraulic fluids.

**G.N.R. S.r.l.** designs and produces X-Ray Diffractometers (XRD) and X-Ray Fluorescence Spectrometers (XRF) for the study of material structure and elemental composition for both academic and industrial applications.

GNR Head Office and Production Site is located in Agrate Conturbia (Novara), near Lago Maggiore; 20 minutes from MALPENSA Airport.

## Certified Company

Highest quality in our products and services is a core value for GNR.
Full commitment is dedicated to support our quality system in the overall process and continuous improvement is fundamental to guarantee GNR compliance to the internationally accepted quality management standard ISO 9001.


CERTIFICATE ◆ CERTIFICATE ◆ СЕРТИФИКАТ ◆ CERTIFICADO ◆ CERTIFICAT ◆ ZERTIFIKAT

CERTIFICATO

Nr 50 100 4258 - Rev. 05

Si attesta che / This is to certify that

IL SISTEMA QUALITÀ DI
THE QUALITY SYSTEM OF

G.N.R. S.r.l.

SEDE LEGALE E OPERATIVA:
REGISTERED OFFICE AND OPERATIONAL SITE:

VIA TORINO 7
I-28010 AGRATE CONTURBIA (NO)

È CONFORME AI REQUISITI DELLA NORMA
HAS BEEN FOUND TO COMPLY WITH THE REQUIREMENTS OF

UNI EN ISO 9001:2008

QUESTO CERTIFICATO È VALIDO PER IL SEGUENTE CAMPO DI APPLICAZIONE
THIS CERTIFICATE IS VALID FOR THE FOLLOWING SCOPE

Progettazione, fabbricazione, assistenza e messa in esercizio di spettometri ad emissione ottica e strumentazione a raggi X per la caratterizzazione dei materiali. Commercializzazione di prodotti associati ed affini (IAF 19, 29)

Design, manufacture, service and start-up of optical emission spectometers and x-ray instruments for materials characterization. Trade of complementary and similar product (IAF 19, 29)

ACCREDIA

Per l'Organismo di Certificazione
For the Certification Body
TÜV Italia S.r.l.

Validità / Validity
Dal / From: 2016-04-26
Al / To: 2019-09-14

Data emissione / Printing Date
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TÜV Italia • Gruppo TÜV SÜD • Via Carlucci 125, Fol. 23 • 20099 Sesto San Giovanni (MI) • Italia • www.tuv.it

GNR periodically organizes at its facility courses and training for technicians and agents as well as seminars and demonstrations.

Thanks to an extensive network of agents GNR provides technical support and delivers spare parts worldwide.

# EUROPE THETA/THETA

## BENCHTOP X-RAY DIFFRACTOMETER


**GNR Analytical Instruments Group** is a worldwide market leader in supplying advanced X-Ray (XRD, XRF) and Optical Emission Spectrometer (OES) systems for complete solutions in structural and elemental analysis.

These analytical methods provide elemental composition of solids and liquids as well as structural parameters of powders, thin films and bulk materials.

**Europe Theta/Theta** is the new GNR Benchtop powder diffraction system for XRD **qualitative** and **quantitative** analysis of polycrystalline materials.

With an achievable peak resolution < 0.04° 2Θ and an angular accuracy within 0.02° over the whole 2Θ angular range, Europe Theta/Theta guarantees the necessary level of performance for the most demanding x-ray diffraction material investigation in a compact and low cost configuration.

## Which Measurements can be performed with EUROPE Theta/Theta?

- Qualitative and Quantitative Crystalline Phase Analysis
- Degree of Crystallinity Calculation
- Polymorph Screening
- Cell parameters, Crystallite size and Lattice strain determination
- Rietveld refinement for structural characterization

## EUROPE Theta/Theta X-Ray Diffractometer Features

**Europe Theta/Theta**, operating at 600 W, offers characteristics and options that provide maximum flexibility in a benchtop set up without quality compromises and with reliable data analysis.

**Compact Size:** its compact size and robust design enable installation on a lab bench and operations in a small space.

**Theta/Theta Goniometer:** GNR has developed an ultra-compact goniometer, adopting stepper motors with optical encoders, to ensure extremely precise angular positioning.

**Optics:** X-Ray beam collimation is obtained by variable slits that guarantee a perfect alignment of the beam in the equatorial plane, while in the axial plane the divergence is limited by interchangeable Soller slits.
Europe Theta/Theta can mount glass or ceramic X-Ray tubes with high reproducibility and stability of focus position.

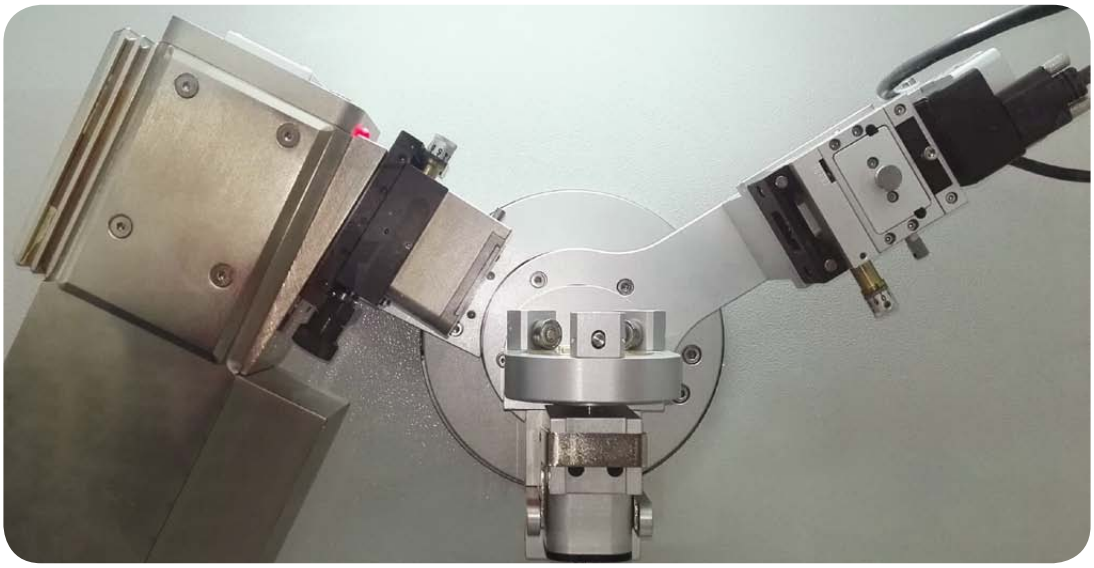
**X-Ray Detectors:** Scintillation counters, silicon drift detectors (SDD), multi strip linear and area detectors (1D, 2D)

**Sample holders:** several sample holders are available to meet the specific needs of each laboratory (rotating sample stage, standard and custom sample holders, air sensitive sample holder, 6 position automatic sample changer).

# EUROPE THETA/THETA

## THETA/THETA GONIOMETER

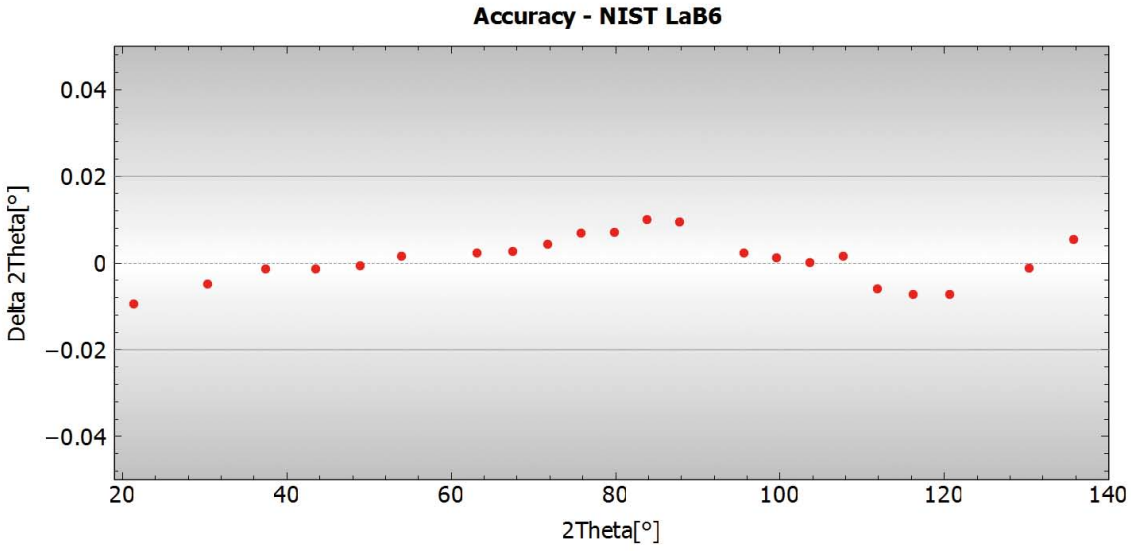
The Theta/Theta configuration allows easy sample preparation, as the measured surface remains in a horizontal plane while the tube and the detector arms are rotating. This configuration is equally suitable for the analysis of loose powders, liquids, thin films and heavy samples that cannot be easily fixed in position.



High-precision goniometer

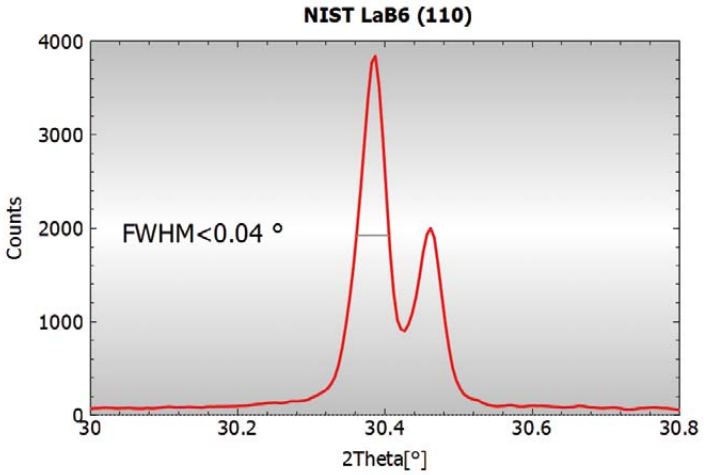
### Goniometer Accuracy

GNR developed a high performance goniometer featuring high precision and outstanding results.
An angular accuracy better than ± 0.02° over the whole 2-Theta range guarantees optimal instrument alignment to support accurate and reliable analysis.



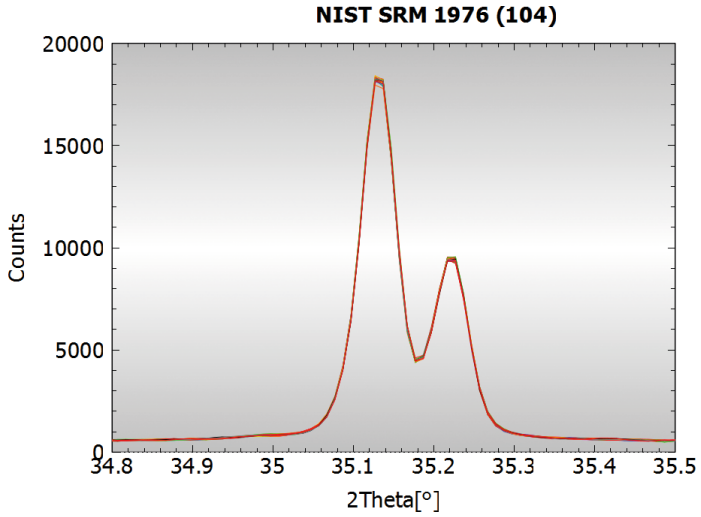

**Setup**
A = 0.5 deg
Soller 2.3 deg
DAS = 0.5 deg
B = 5 mm
Soller 2.3 deg
Ni filter 0.02 mm
SA = 1 deg
Step size = 0.2 deg
Interpolation = 0.01
Linear mode

## Goniometer Resolution




**Setup**
A = 0.1 deg
Soller 2.3 deg
DAS = 0.5 deg
B = 5 mm
Soller 2.3 deg
Ni filter 0.02 mm
SA = 0.05 deg
Step size = 0.004 deg
Interpolation = 0.005
Linear mode

## Goniometer Reproducibility




**Setup**
A = 0.5 deg
Soller 2.3 deg
DAS = 0.5 deg
B = 5mm
Soller 2.3 deg
Ni filter 0.02 mm
SA = 1 deg
Step size = 0.1 deg
Interpolation = 0.01
Linear mode
**StdDev[°]= 0.0002**

25 sequential measuraments with Corundum 1976 (104) peak

A = divergence slit – DAS = divergent beam anti-scatter slit – B = anti-scatter slit

Europe Theta/Theta is pre-aligned at delivery. Every single instrument must pass strict internal test procedures based on the internationally accepted NIST Standard Reference Materials (SRM) 1976 (Alumina), 660 (Lanthanum Hexaboride) or 640 (Silicon).

**Safety Assurance**
**Europe Theta/Theta** complies with the complex statutory requirements regarding x-ray, machine and electrical safety.
Maximum x-ray safety with radiation level significantly below the annual dose limit for general public (1 mSv/year).
The radiation enclosure door cannot be opened when x-rays are on and the system immediately switch off if shutter is forced to open.
This function completely protects users from radiation exposure.

## CeleriX 1D Hybrid Photon Counting (HPC) microstrip detector

CeleriX multi strip detector takes full advantage of both Hybrid Photon Counting (HPC) and Single Photon Counting technologies, providing:

- X-Ray direct detection;
- noise-free performances;
- high intensity measurements;
- extremely fast data collection, without any sacrifice in data quality and peak resolution.

Our CeleriX 1D detector allows to perform data collection in an extremely short time: it can capture simultaneously a large angular range reducing measurement time from hours into minutes.

CeleriX 1D mod. Advanced

CeleriX 1D detector can be used in 0D-mode and turned by 90° to cover an extremely large dynamic range.

| Main Specifications | CeleriX 1D mod. Advanced | CeleriX 1D mod. Plus |
|---|---|---|
| Number of counting pixels | 640 | 1280 |
| Pixel width [μm] | 50 | 50 |
| Active area | 256 $mm^2$ | 512 $mm^2$ |
| Dynamic count rate range [counts/s] | Up to $1x10^{10}$ | Up to $1x10^{10}$ |
| Energy range [keV] | 5-40 | 5-40 |
| Cooling | Air | Air |

## Blitz - Silicon Drift Detectors (SDDs)

One of the main influences on the quality of X-ray diffraction experiments is the level of background.
The sample itself contributes to the overall background level particularly when the incident beam causes it to emit secondary X-rays (i.e. fluorescence).

The Blitz energy dispersive detector, thanks to its excellent energy resolution, is the ideal means of removing these unwanted background features.

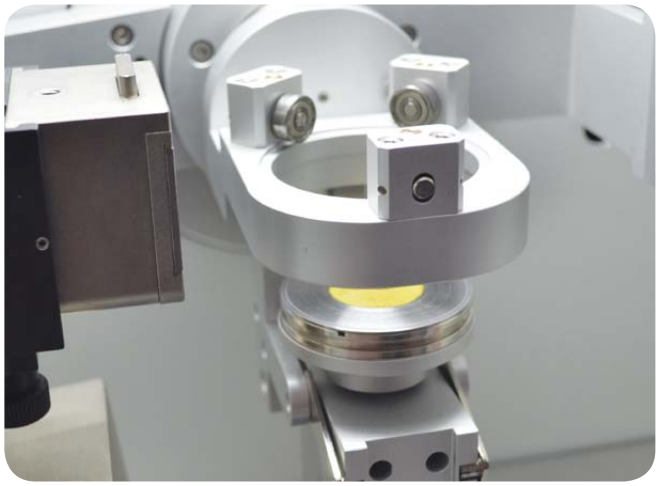
GNR Silicon Drift Detectors (SDDs) combine a large sensitive area with a small value of the output capacitance and are therefore well suited also for high resolution, high count rate X-ray spectroscopy like EDXRF.

| Main Specifications | Silicon Drift Detector - SDD |
|---|---|
| Active area | 5 – 100 $mm^2$ |
| Shaping time | Standard: 1 μs or 250 ns |
| | Customizable: 10 μs - 100 ns |
| Energy resolution | Shaping time 1 μs: 124 eV FWHM@Mn Kα |
| | Shaping time 250 ns: 136 eV FWHM@Mn Kα |
| Cooling | Air |


Blitz - Silicon Drift Detectors

## Rotating sample stage

Rotating sample stage enhances measurement quality for sample with non-randomly oriented crystallites.



Rotating sample stage

## Multi sample holder

Automatic sample positioning for long-time analysis without user attendance.
Each position has a built-in rotating stage (option).

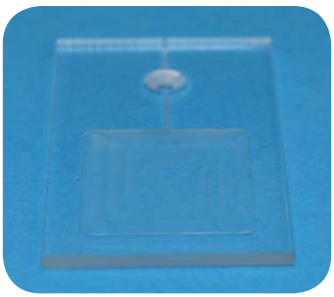

6-position auto-changer

## Specimen holders

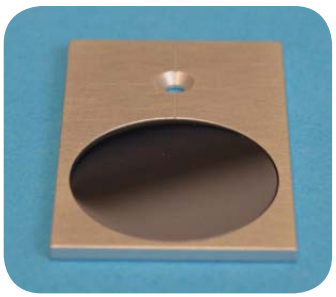
**GNR** provides several dedicated solutions for different kinds of specimen. These fit the analytical requirements and guarantee the best achievable data quality. Here below an overview of some specimen holders available. Upon request tailored solutions can be realized to further optimize the analytical results.

**Flat specimen holders (length 44 mm)**

Front loading sample holder
Material: Al
Cavity:
HxL: 20 mm x 20 mm
Depth: 1 mm



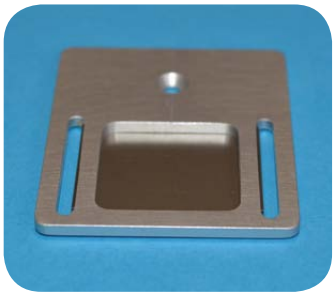
Front loading sample holder
Material: PMMA
Cavity:
HxL: 20 mm x 20 mm
Depth: 0.2/0.5 mm



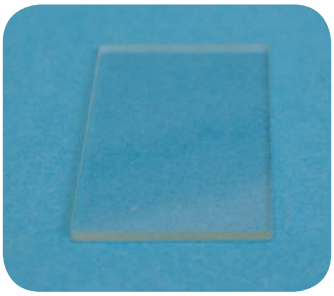
Si zero background sample holder
Material: Al
Cavity:
Diameter: 24.7 mm
Depth: 0.5/0.1 mm

# EUROPE THETA/THETA

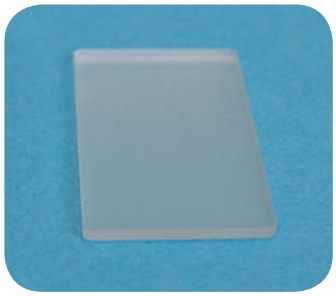
## SAMPLE HOLDERS



Back loading sample holder
Material: Al
Cavity:
HxL: 20 mm x 20 mm
Depth: 1.9 mm

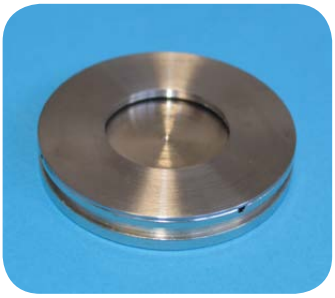


Glass slide for clay oriented mount

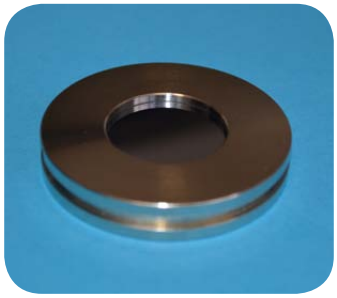


Glass slide for side loading sample holder filling

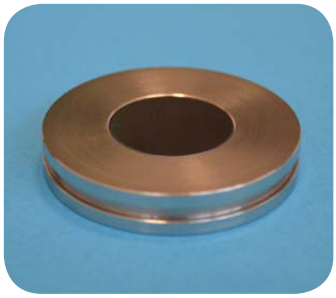
### Circular sample holders (external diam: 51.5 mm - Herzog press)



Back loading sample holder
Material: steel
Cavity:
Diameter: 24.7 mm
Depth: 2.5 mm

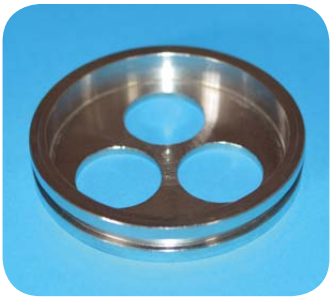


Side loading sample holder
Material: steel
Cavity:
Diameter: 24.7 mm
Depth: 2 mm

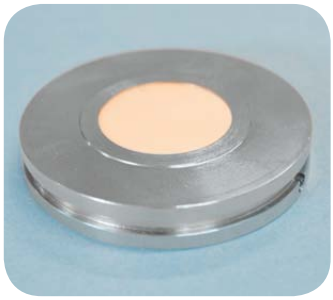


Si zero background
sample holder
Material: steel
Cavity:
Diameter: 24.7 mm
Depth: 0.2 mm

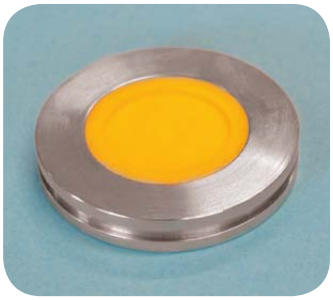
Clay oriented aggregate mount
sample holder
Material: steel and glass
Cavity:
Area: 37 x 37 $mm^2$
Depth: 2.5 mm
Glass: 25x25 $mm^2$



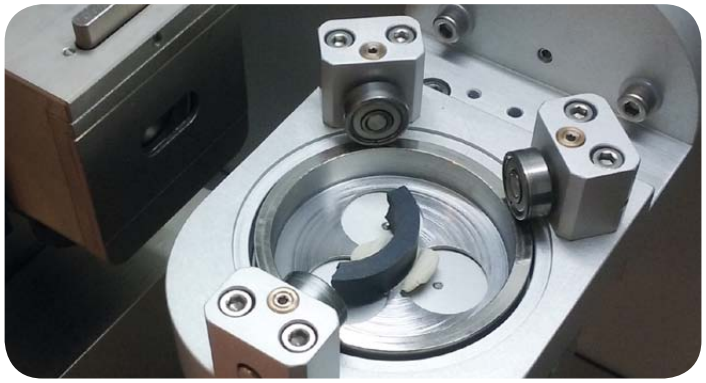
Massive sample holder
Material: steel
Cavity:
Diameter: 45 mm
Depth: 7 mm



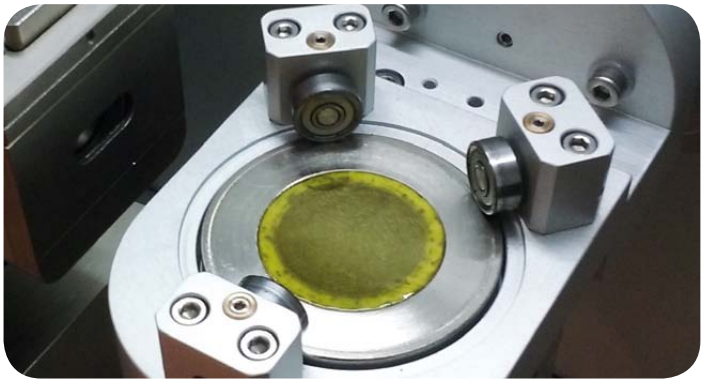
Air monitoring filter sample holder
Material: steel
Cavity:
Diameter: 25.7 mm



Air sensitive sample holder
Material: steel and teflon
Cavity:
Diameter: 25 mm
Depth: 1.5 mm
Foil: Kapton, 8 µm



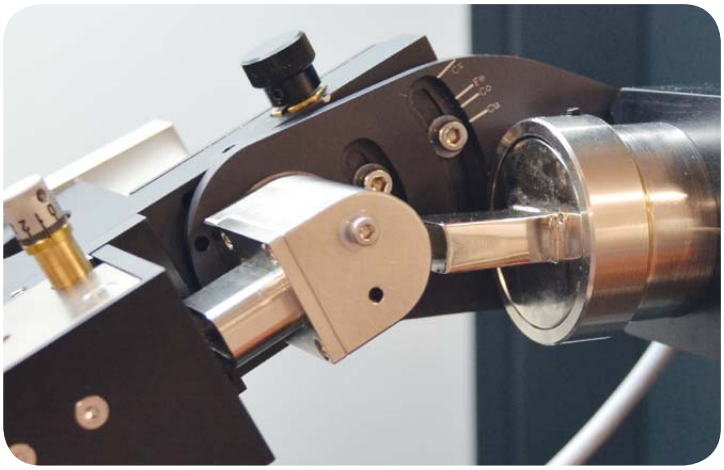
Massive sample holder



Air sensitive sample holder

## Secondary graphite monochromator

Secondary graphite monochromator removes the continuous component of the spectrum, Kβ as well as fluorescence from the sample. It ensures an excellent signal to noise ratio.



Secondary graphite monochromator

## Non-ambient X-ray diffraction

Non-ambient X-ray diffraction has become an indispensable technique to understand the influence of temperature, atmosphere or pressure on materials of any kind. Besides its relevance for conducting research, this knowledge is essential for optimizing technical processes and performing quality control in industrial applications.

The BTS 150/500 benchtop heating stages are the first commercial non-ambient stages for benchtop diffractometers.

Both stages are extremely compact in design to fit into the restricted space of typical benchtop diffractometer. The control electronics is integrated in the heating stages and provides easy operation.


BTS 500

**BTS 150/500**

**Technical data**

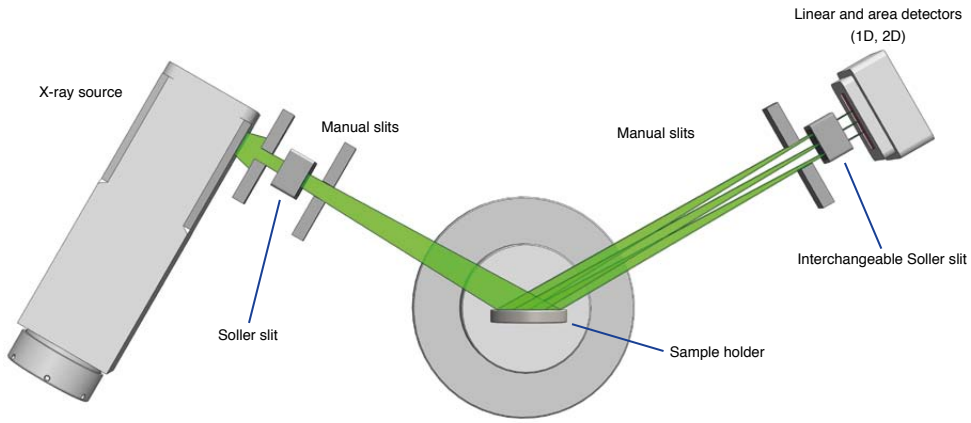
| | |
|---|---|
| Temperature range | -10 °C to 150 °C (BTS 150)<br>Ambient to 500 °C (BTS 500) |
| Atmospheres | Air, inert gas, vacuum ($10^{-1}$ mbar) |
| X-ray geometry | Reflection |

# EUROPE THETA/THETA

## APPLICATIONS

Phase analysis is the study of the different polycrystalline materials within a sample. One phase is identified among the others due to its unique powder diffraction pattern which arises from its unique combination of composition and crystal structure.

The analysis is applicable to all types of crystalline materials and can be restricted to identification only or extended to full quantitative analysis.




**EXPLORE POWDERS**

- Crystalline phases ratio (E. Calibration, RIR, Rietveld)
- Amorphous content in samples
- Crystal structure
- Phase transitions
- Crystallinity index and percentage

**GNR Europe Theta/Theta** fits well customer analytical needs in many fields:

- Geology and Mineralogy
- Clays
- Glass - Ceramics
- Cements
- Petrochemicals
- Catalysts
- Polymers
- Forensics
- Agricultural Sciences
- Biosciences
- Chemicals
- Pharmaceuticals
- Cosmetics
- Enviromentals
- Art and Archeology

Geology and Mineralogy
Determine the composition of raw material

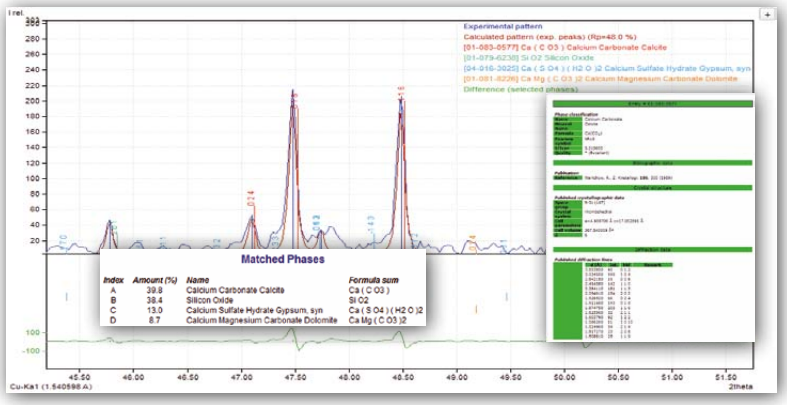
Petrochemicals
Analysis of solids obtained during the drilling process

Chemicals
Characterize unknown materials and support R&D

Pharmaceuticals
Quality control, identification and polymorphs screening

## Phase Identification and Quantitative - Mineralogy

**GNR Europe Theta/Theta** can be used for phase identification and quantitative analysis in mineralogy domain. Match! can identify the phases present in the sample and report the weight percent composition.
In this case the sample is a mixture of Quartz, Calcite, Gypsum and Dolomite.

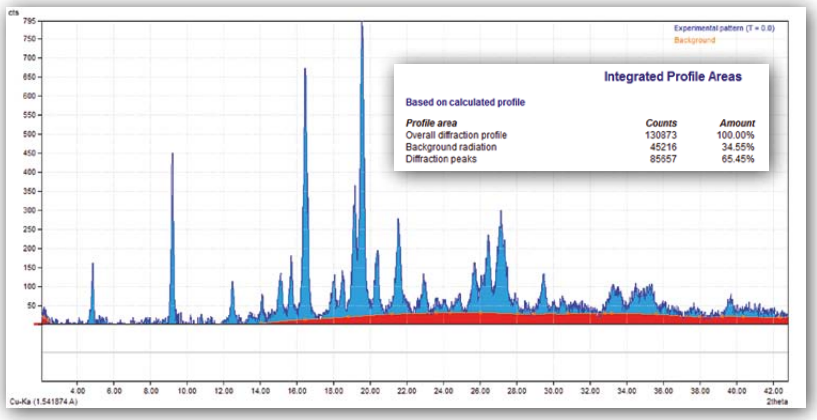



Quantitative Phase Analysis using Match!

## Crystallinity Degree Calculation - Pharmaceutical

In pharmaceutical industry product development and quality assurance steps, it is common to measure the amorphous phase amount in order to determine the degree of crystallinity.

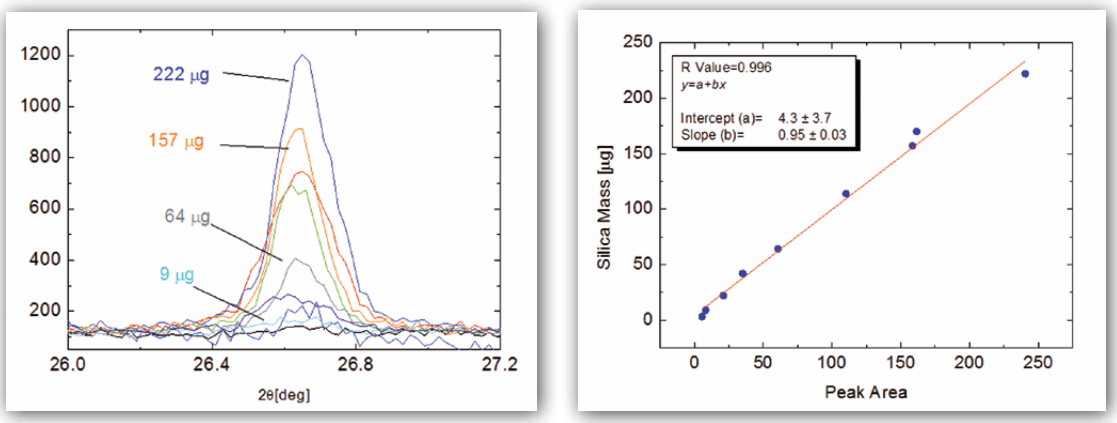
It is important to monitor the amount of the amorphous phase within a drug because of its thermodynamic instability, relative to the crystalline state. Match! allows to determine it easily through the Degree of Crystallinity calculation.




Crystallinity Degree Calculation using Match!

## Silica Dust Monitoring - Environmental

Crystalline silica is a basic component of soil, sand, granite, and many other minerals. Quartz is the most common form of crystalline silica. The seriousness of the health hazards associated with silica exposure is demonstrated by the fatalities and disabling illnesses that continue to occur in sandblasters and rock drillers. Crystalline silica has been classified as a human lung carcinogen. Measuring and quantifying silica dust collected on air filter is important to protect health of workers involved in activity exposed to silica dust.
Quartz (101) diffraction peak area is proportional to the mass of silica dust deposited on a filter. Using NIST – SRM 1878 quartz standard is possible to calculate a calibration curve for silica dust mass quantification and monitoring.



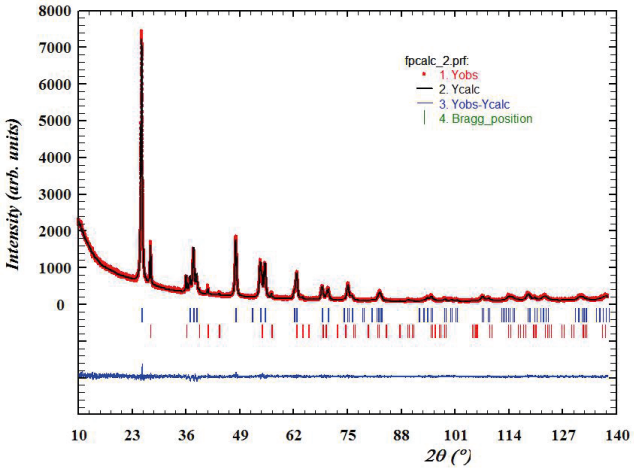

Calibration curve for silica dust mass quantification

## Crystallite size estimation of nanopowders

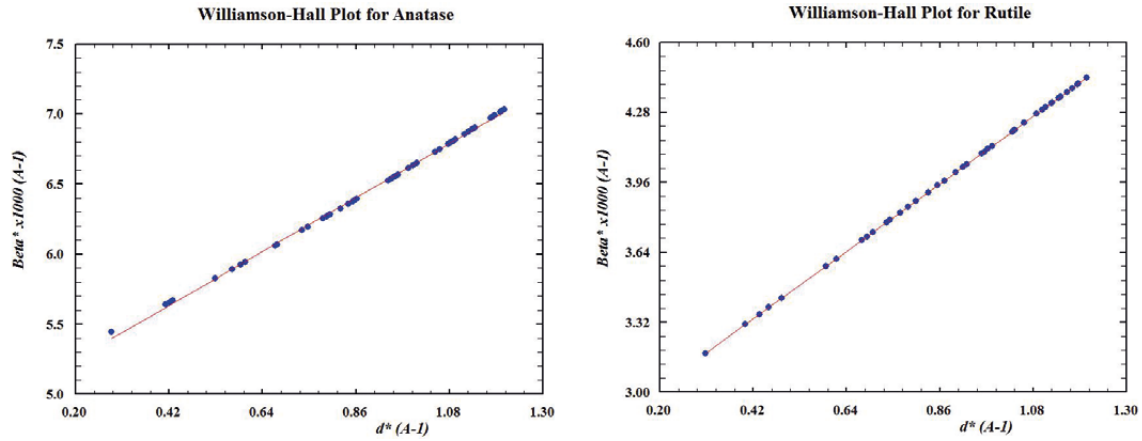
Nanocrystalline materials are currently an active field of research: the possibility of tailoring their functional properties, by modifying their microstructure, makes them very appealing. XRD is an effective technique to study their defective microstructure through Line Profile Analysis: Europe 600 benchtop diffractometer equipped with Celerix linear detector allows to perform Rietveld refinement with extraction of microstructural parameters in a fast and easy way.

After instrumental broadening determination by means of NIST SRM 660 (LaB6), a nanocrystalline mixture of Rutile and Anatase (NIST 1898) was analysed and apparent average crystallite size for both phases was estimated by simple Scherrer formula (MATCH!) and integral breadth methods implemented in FullProf Rietveld program[1].

[1]J. Rodríguez Carvajal and T. Roisnel. Materials Science Forum 443-444, 123-126 (2004).




Rietveld refinement plot for Anatase (blue) and Rutile (red) mixture.




W-H plots for Anatase and Rutile. Lorentz-Lorentz approximation is assumed

| Apparent crystallite size [nm] | | | | |
|---|---|---|---|---|
| **Phase** | **Peak** | **Match!** | **Match! Uncor-rected** | **Certificate Information Value** |
| Anatase | (200) | 23.6 | 23.3 | 23.6 |
| Rutile | (111) | 48.6 | 39.3 | 44.1 |

Results of Scherrer analysis for selected peaks

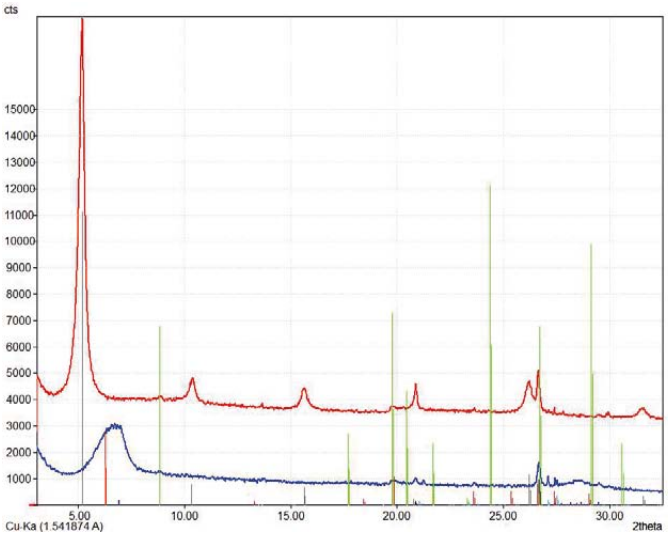
| Volume weighted apparent crystallite size [nm] | | | |
|---|---|---|---|
| **Phase** | **FullProf** | **W-H Plot** | **Certificate Information Value** |
| Anatase | 20 | 20 | 19±2 |
| Rutile | 36 | 36 | 37±6 |

Results from FullProf microstructural analysis

## Oriented aggregate mounts for Clay minerals identification

The correct identification of clay minerals is very important[1]: the clay fraction is separated from the sample and prepared as an oriented aggregate on glass slides to enhance diagnostic basal reflections signal. The corresponding d-spacing contraction or expansion during treatments allows a clear identification, especially if layer interstratification is present. Typical treatments ar: air drying, glycolation with ethylene glycol, heating to 400 °C and 550 °C. Diffraction pattern collection must start at low angles ($2\theta < 5$ deg.) in order to collect basal reflections at very large d-spacings (e.g. Smectite, Chlorite groups)[2].

In the example reported below a Bentonite sample (SWy-2) was deposited directly as an oriented aggregate on a glass slide. First air drying and then ethylene glycol treatments were performed. Diffraction pattern were collected from $2\theta$ 3 to 35 deg by **GNR Europe Theta/Theta** diffractometer with Cu FF anode, Ni filter and CeleriX 1D mod. Advanced 640 linear detector.




The blue pattern represents the air dried sample: the red vertical bar corresponds to Na-Montmorillonite phase 001 peak (d~14 Å) with different water layers (1 to 2), while the green one the Illite 001 (d~10 Å). Quartz (101) peak is visible at 26.64 deg, while Feldspars are detected in 27.1-27.5 deg region.

The clear difference after glycolation (red pattern) is the enhanced signal of the Montmorillonite basal peak and its shift to larger d values (~17 Å), which is compatible with a glycolated-Montmorillonite clay mineral (grey bar), according to ICDD PDF4 and USGS Clay Mineral Identification Flow Diagram[3]. Higher orders (002, 003, 004, 005 and 006) are visible now too. Illite is unaffected by glycolation, as expected.

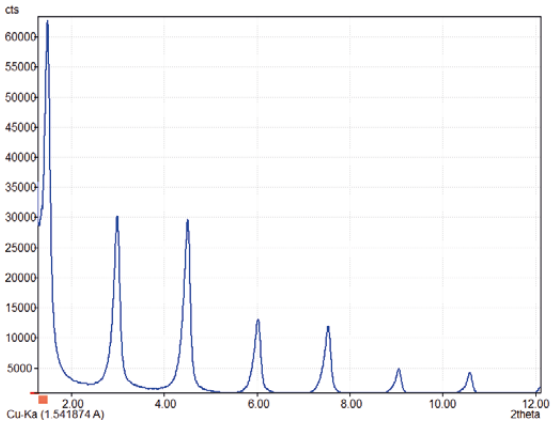
[1] F. Bergaya, B.K.G. Theng and G. Lagaly, Handbook of Clay Science, Developments in Clay Science, Vol. 1 2006 Elsevier.

[2] A laboratory Manual for X-Ray Powder Diffraction, USGS, http://pubs.usgs.gov/of/2001/of01-041/

[3] https://pubs.usgs.gov/of/2001/of01-041/htmldocs/flow/start.htm

**Performances at low scattering angle: Silver behenate**

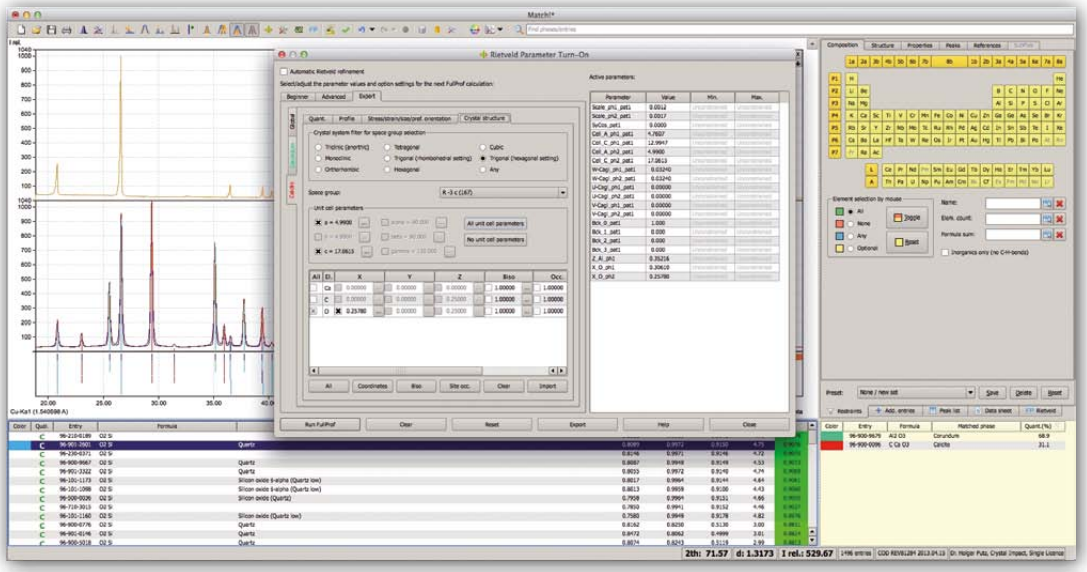
Configuration:
Divergence slit = 0.5 deg
Anti scatter slit = 0.5 deg
Soller slits = 2.3 deg
Detector slit = 2.2 mm
CeleriX 1D mod. Advanced Active area = 0.8 deg

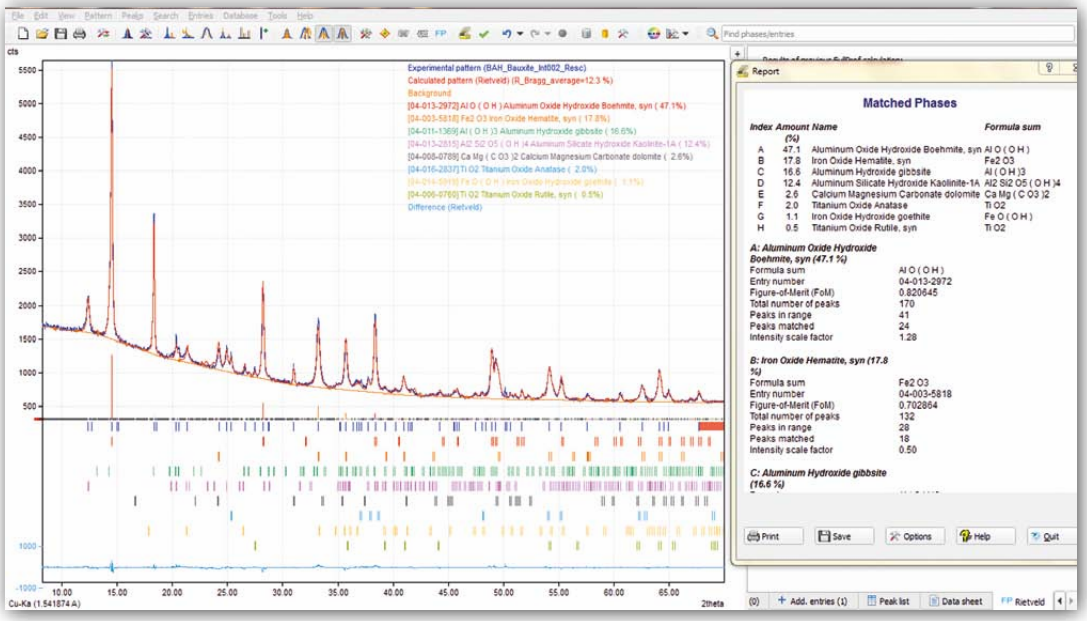


## Software

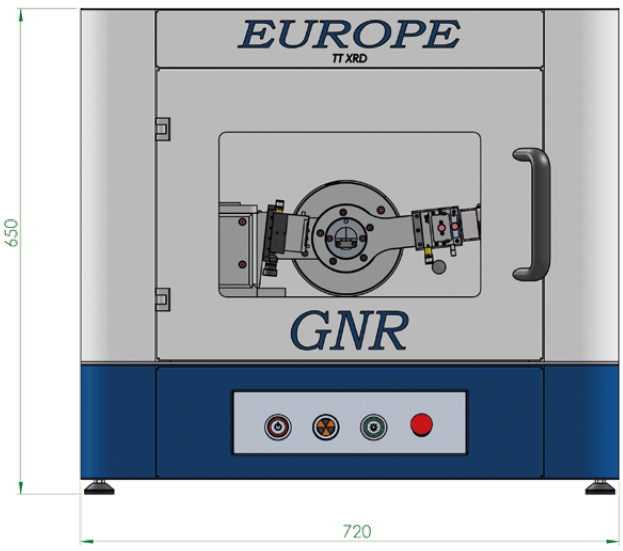
**GNR Europe Theta/Theta** adopts a specific modular design software package able to support the user in all activities.
GNR software supports several type of analysis, from Data Acquisition, having the full control of all the process and hardware settings (motors, x-ray generator and tube, detector, measurement set up), to Data Analysis.

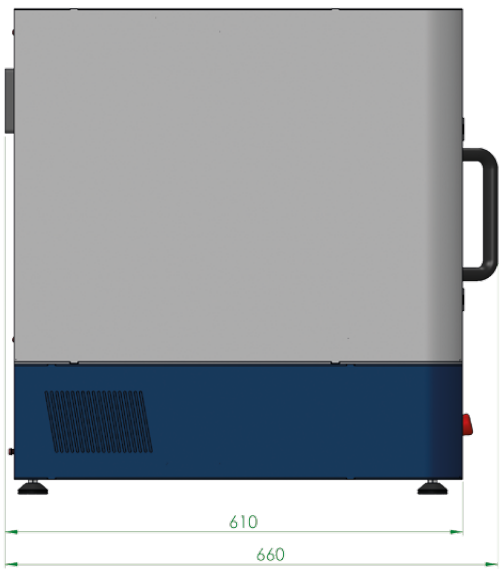
Match! is an easy-to-use software for phase identification from powder diffraction data.
It compares the diffraction pattern of your sample to a database containing reference patterns in order to identify the phases present in the sample. Additional knowledge about the sample like known phases, chemical elements or density can be applied.
In addition to this qualitative analysis, a quantitative analysis (using either RIR method or Rietveld refinement) can be performed as well.
End user can easily setup and run Rietveld refinements within Match!, with the actual calculations being performed automatically, using the well-known program FullProf in the background. Match! provides a gentle introduction into Rietveld refinement, from fully automatic operation to the “Expert” mode.
As reference database, you can apply the included free-of-charge COD database and/or ICSD/Retrieve (if you have a valid license), use any ICDD PDF product, and/or create a user database based on your own diffraction patterns.

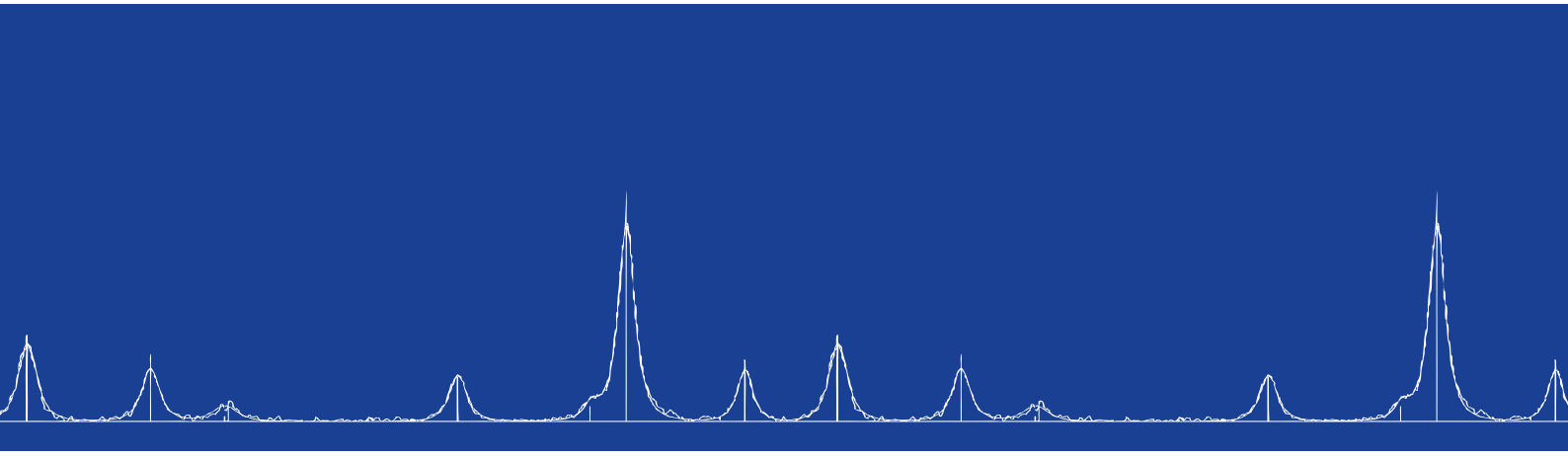




| | |
|---|---|
| **Configurations** | Theta/Theta geometry |
| **Measuring circle radius** | 160 mm |
| **Scanning angular range** | - 5° < 2-Theta < + 160° (depends on accessories) |
| **Angle positioning** | Stepping motors with optical encoders |
| **Angular accuracy** | Better than ± 0.02° over the whole 2-Theta range |
| **Angular resolution** | < 0.04 2-Theta on $LaB_6$ (110) peak |
| **X-ray generator** | 600 W |
| **Max. output voltage** | 40 kV |
| **Max. output current** | 15 mA |
| **X-ray tube** | Glass (option ceramic), Cu anode<br>(option: Co, Fe, Cr, Mo, W, Ag) |
| **Focus** | 0.4x8 mm FF (Fine Focus)<br>(options: 0.4x12 mm LFF; 1x10 mm NF; 2x12 mm BF) |
| **Divergence Slit** | Variable 0-4° |
| **Scattering Slit** | Variable 0-4° |
| **Receiving Slit** | Variable 0-6 mm |
| **Interchangeable Soller Slits** | 2.3° (standard)<br>(options: 1°, 2°, 4°) |
| **Detectors** | Scintillation counter<br>CeleriX 1D/2D multi strip detectors<br>Silicon Drift Detectors (SDDs) |
| **External dimensions** | 720 mm, 650 mm, 660 mm |
| **Weight** | 120 Kg |
| **Cooling water supply** | External Compact Low Noise Version |
| **Power Supply** | 90 - 250 Vac, single phase |
| **Maximum power consumption (including water chiller)** | 2 kVA |





Local Agent

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