Thermo Scientific ARL X'TRA Powder X-ray Diffraction System

Uncompromised Intensity and Resolution

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ARL X'TRA
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High performance powder X-ray diffraction system

The Thermo Scientific ARL X'TRA is an advanced multi-purpose system designed for academic or industrial analytical laboratories. It is ideal either for routine analysis or highly demanding applications requiring dedicated accessories. Analytical performance, ergonomics and safety are the major characteristics of this outstanding instrument.

With 20 years of experience in the North America market and high precision engineering with excellence in X-ray spectroscopy, the ARL X'TRA offers a leading edge in the field of X-ray powder diffraction technology.

The design of the ARL X'TRA is based on a vertical θ-θ Bragg-Brentano geometry for convenient sample preparation and sample handling. With the unique Peltier detector technology, ultimate performance in both angular and energy resolution can be achieved without using β-filters or monochromators. This elegant design yields higher diffraction intensities and superior resolution compared to traditional point detectors.

Depending on the type of analysis or sample type, the system can be easily reconfigured using pre-aligned Plug & Play accessories such as temperature chambers, parallel beam optics, transmission stage.

The modular approach of the ARL X'TRA powder diffraction instrument provides full flexibility without any compromise in performance for a large range of applications covering both inorganic and organic polycrystalline materials.

A wide range of analytical techniques

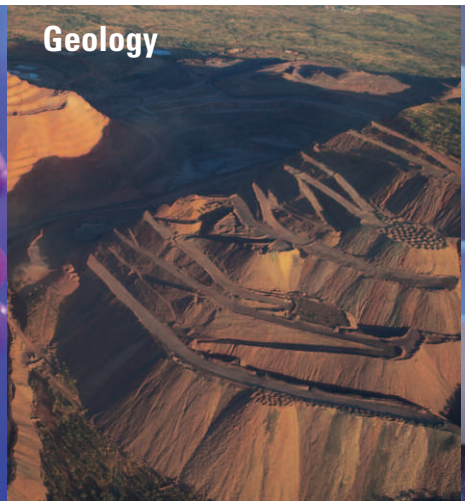
X-ray diffraction is a versatile and non-destructive technique that reveals detailed structural and chemical information about the crystallography of materials.

- Identification of single or multiple phases in an unknown sample
- Quantification of known phases of a mixture
- Crystallography – solving crystal structure
- Non ambient analysis – crystal structure changes with temperature, pressure or gas phase
- Surface and thin film analysis
- Texture analysis

A broad range of materials

Natural or manufactured materials of organic or inorganic chemistry can be characterized with this technique. Some typical materials are listed here:

- Chemicals
- Pharmaceutical substances
- Environmental dusts
- Asbestos
- Rocks
- Clay minerals
- Zeolites
- Polymers
- Metals
- Cement and building materials
- Forensics
- Pigments

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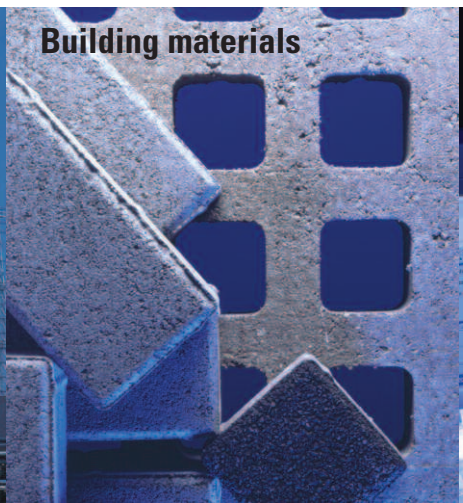
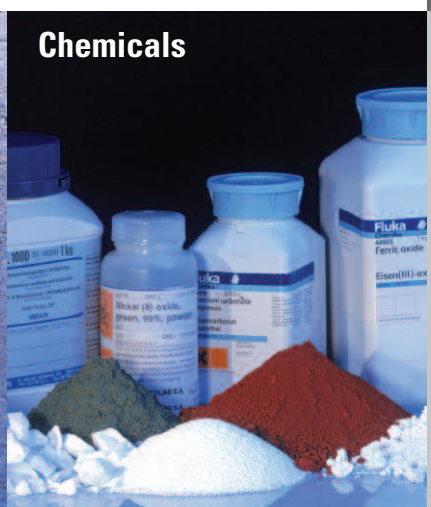
Environment


Building materials


Chemicals
Fluka
Ferric oxide

Technique

The most flexible goniometer geometry

It is now well accepted that for the majority of the powder X-ray diffraction experiments, the vertical θ-θ Bragg-Brentano setup replaces the traditional vertical or horizontal θ-2θ geometry. The main reason is that the sample stage does not move during the scan and therefore even loose powders or liquids can be analyzed.

In this geometry, the ARL X'TRA offers the best configuration providing superior flexibility for future upgrades or reconfigurations with optional accessories.

The reliability and accuracy of this system is achieved thanks to closed loop servo motors with optical encoders.

Choose the best trade-off between intensity and resolution

The continuous slit aperture capability of the ARL X'TRA system allows an instrument operator to choose the optimum balance between intensity and angular resolution.

Continuous adjustment capability allows instrument operators to optimize the slit settings in accord with the crystallographic quality of the materials.

As shown in Figure 3, an angular resolution < 0.04° FWHM can be measured easily in standard configuration without using beam path accessories and with a normal scan speed (0.1° 2θ/s).

A precise control of the radial and axial collimation of the beam is ensured with micrometer-controlled slits and an exchangeable/removable set of soller slits.

Precise "zero plane" adjustment

Sample height is critical to get accurate data in Bragg-Brentano geometry (standard setup) and to get best intensity in parallel beam configuration.

The standard sample stage on the ARL X'TRA includes a micrometer based stage with height adjustment and height position block. Depending on the shape and dimension of the sample, the micrometer adjustment can be used to precisely adjust the specimen height at the scale of 10 µm. The precision in the micrometer adjustment also yields numeric traceability of the aligned position.

Modular stage design

The modular design or so called "Plug & Play" of the ARL X'TRA allows users to exchange pre-aligned sample stages without requiring a lengthy realignment procedure. The standard sample stage incorporates one mechanical pin that precisely orient both the translational and rotational orientation of the stage relative to the goniometer. Complete reconfiguration of the diffractometer is now achievable in several minutes instead of hours.

Unique Peltier cooled Si(Li) solid-state detector

For more than 20 years, solid-state detectors have been a popular detection option on our instruments. The latest version is a Peltier cooled Lithium drifted Silicon solid-stage detector (see Figure 1). It is sealed under high vacuum with passive getters that ensure the long term stability of the vacuum.

The 5 stage Peltier pyramid allows to cool down the Si(Li) crystal down to a temperature close to -100°C and therefore, the internal noise of this detector is extremely low: <0.1 CPS.

Quantitative phase analysis in the range of 0.1 weight % (depending on the sample) is possible due to very low detector noise level.

This solid-state detector allows the user to electronically select photons based upon their energy signature thereby eliminating the need for beta filters or diffracted beam monochromators to remove unwanted source of X-ray intensity (e.g. diffraction from beat wavelengths, Bremsstrahlung and/or sample fluorescence as shown in Figure 2).

The fluorescence efficiency for Fe with Cu radiation is very high so if we look at a Fe powder, a conventional scintillation detector shows about 5,000 cps of Fe fluorescence. It is also showing Kβ contributions for the stronger diffraction peaks (green curve in Figure 2). This is a big problem with conventional detectors because while the β filter can get rid of Kβ, Fe fluorescence cannot be discriminated. The only way to get rid of the Fe fluorescence background is to use secondary beam monochromators but this strongly reduces overall intensities. The Si(Li) Peltier detector has no problem removing these interferences.

This resolution in energy can also be exploited to tune the wavelength and therefore the energy. To get simplified XRD pattern with minimum peak overlaps, one can for instance collect a pattern in Kβ mode instead of Kα which shows its characteristic Kα1/2 doublet. The optical mount design allows both a para-focussing slit optic and thin film collimator optic to be mounted in aligned positions simultaneously. The detector is mounted on a double pinned bracket that allows it to be moved from behind one optic to the other. This design feature allows it to switch to optical configurations in less than five minutes without having to re-align the instrument. In addition, this solid-state detector produces superior quality XRD pattern in both para-focusing and parallel beam optics. There is no compromise in resolution or quality of XRD data when parabolic mirrors are employed.

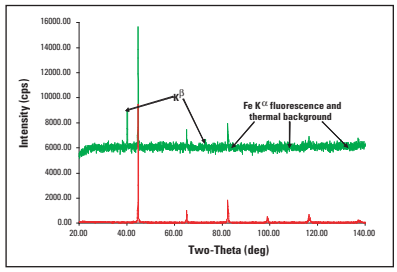


Figure 2: Two measurements of iron powder showing the effect of sample fluorescence suppression and Kβ removal with Peltier detector. All peaks have the same net intensity.

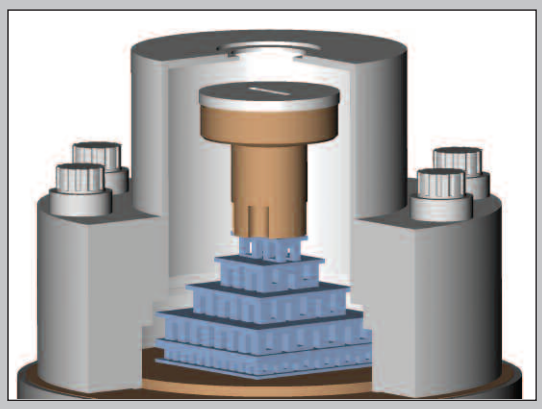
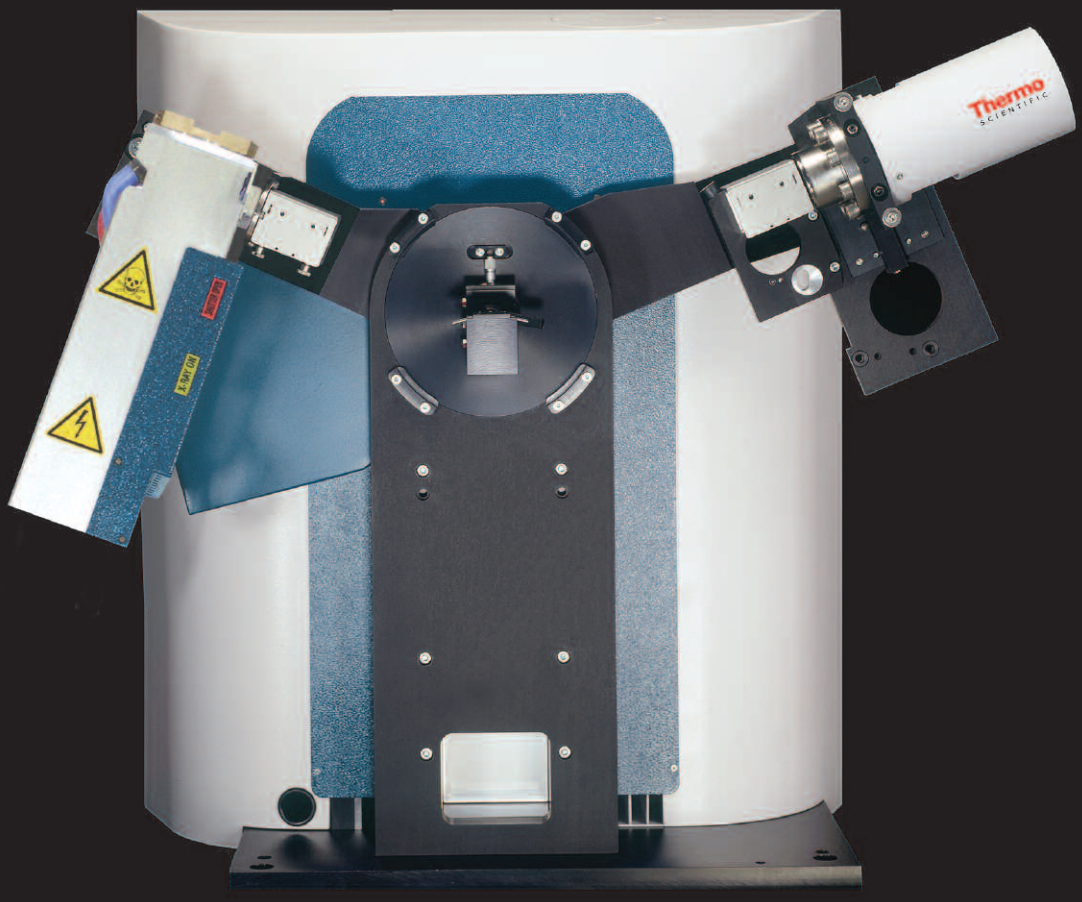
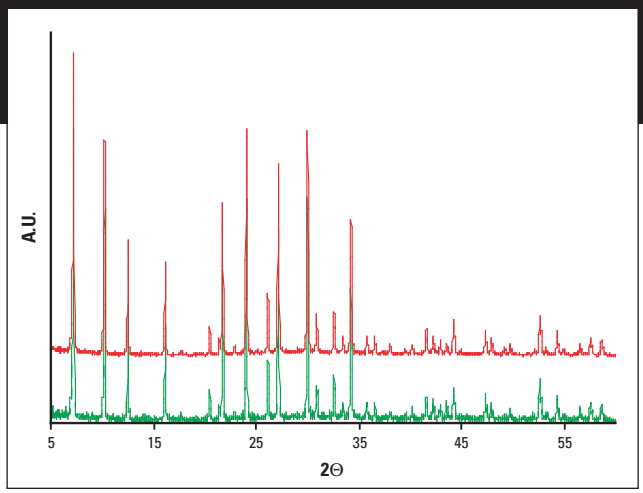


Figure 1: Cross section of the sealed head of the Peltier detector

The unique design of the Peltier detector is absolutely maintenance free and has a long term stability guarantee





Speed of acquisition with the ARL X'TRA on a Zeolite 4A sample: In green, the data measured in less than 5 minutes and in red, the data measured in 20 minutes

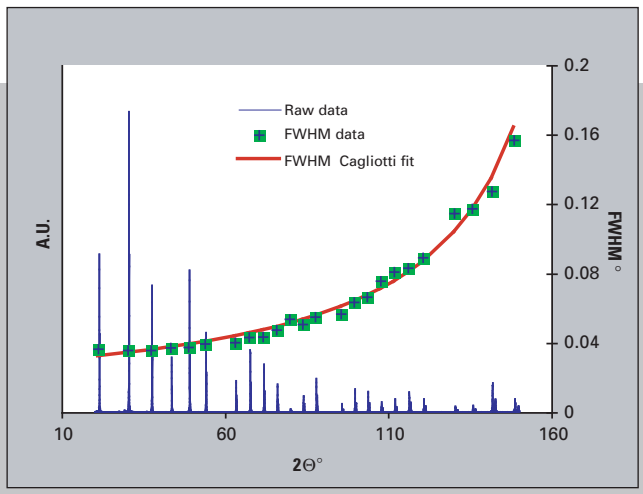


Figure 3: Full Width at Half Maximum (FWHM) of the NIST LaB6 (SRM660a) reflections as a function of 2θ CuKα. The red curve shows the Cagliotti fit and in blue, the XRD raw data measured in 2 hours without primary or secondary beam monochromator

WinXRD for data acquisition and data analysis

Best performance from the X-ray diffraction system is ensured with WinXRD, a Windows® XP based software. This comprehensive software package is fully integrated and facilitates a smooth progression from data collection through analysis and report writing.

Data can be handled with convenience and efficiency thanks to audit trails, user access controls and export functions.

The data collection part offers the possibility to perform different types of measurements:

- Symmetric θ:θ normal scans
- Asymmetric θ scans (e.g. for grazing incidence)
- And finally ω rocking scans.

Data analysis software includes various analytical features like:

- Basic data treatment (background substraction, smoothing, peak extraction, profile fitting, etc.)
- Search-match routine using the ICDD or user database
- Quantitative Phase Analysis
- Crystallite size determination
- Crystallographic analysis (indexing and unit cell refinement)
- 2D and 3D graphic display for multiple graphs
- Texture analysis with 2D and 3D polefigure display

Advanced third party software allow more specific data analysis like:

- SIROQUANT for routine minerals quantification based on the Rietveld method
- Visual CRYSTAL® for full pattern qualitative and quantitative analysis.

Enhanced capabilities with accessories

In addition to the superior performance of the standard instrument configuration mainly dedicated to phase identification & phase quantification, the system can be quickly reconfigured with accessories providing superior analytical capabilities like:

- Batch mode for routine measurements with the multi-position sample changer
- Texture analysis in transmission mode with the transmission stage
- Non-ambient experiments from low temperature to very high temperature conditions under vacuum, air, inert gases or controlled humidity conditions
- Air sensitive material analysis using the capillary stage
- Reactive materials with the environmental stage
- Low Z materials, small samples, irregularly shaped samples can all be characterized with great efficiency using the parallel beam setup consisting of the parabolic mirror and the thin film collimator and more.

Rapid & reproducible accessory exchange

Recent developments in high precision mechanics significantly enhanced the positioning reproducibility of the accessories. With the 'Plug & Play' system, it is now possible to quickly exchange stage accessories and optical configurations without requiring time consuming re-alignment. Moreover, the software configuration manager stores all specific settings so that the user only has to select the pre-aligned optical and stage accessories currently installed from software menus. All instrument configuration details are recorded and associated with the data.

From the left to the right (Figure 1), the exchangeable components on the goniometer are:

- X-ray tube
- Primary beam path accessories: divergent slits, soller slits, anti-scatter slits, parabolic mirrors, pinhole collimator, filters
- Sample stages: basic stage, single sample spinner, sample changer, environmental chamber, capillary stage, transmission stage, temperature stages (low temperature to high temperature), pressure reactor
- Secondary beam path accessories: anti-scatter slits, soller slits, receiving slits, thin film collimator, monochromators
- Detectors: Peltier cooled solid-state detector or Scintillation detector

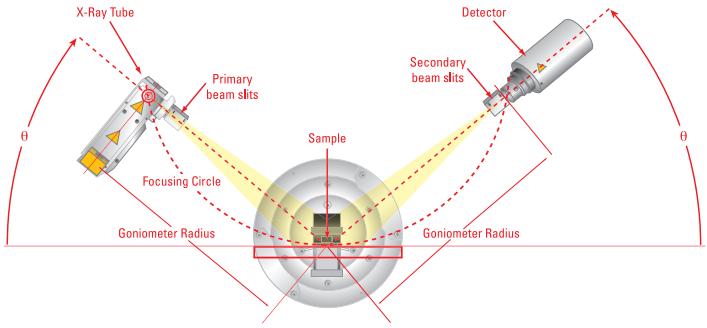
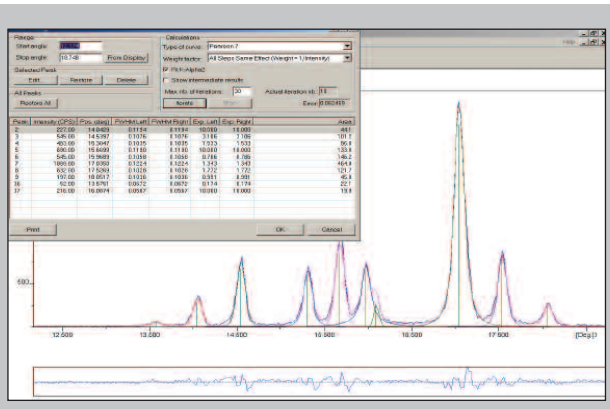
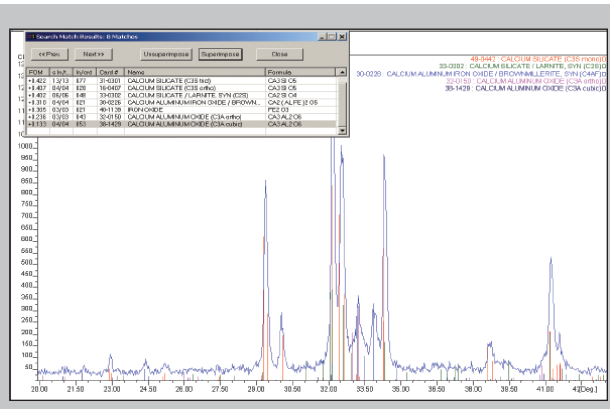


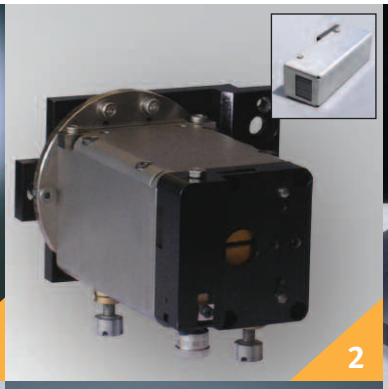
Figure 1: Schema of the geometry of the theta:theta goniometer

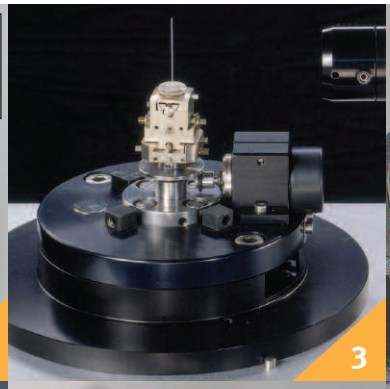


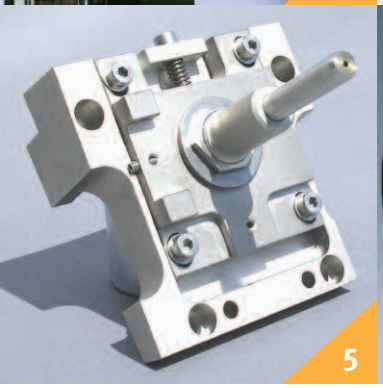
Peak position extraction by profile fitting, an example of Ranitidine analysis



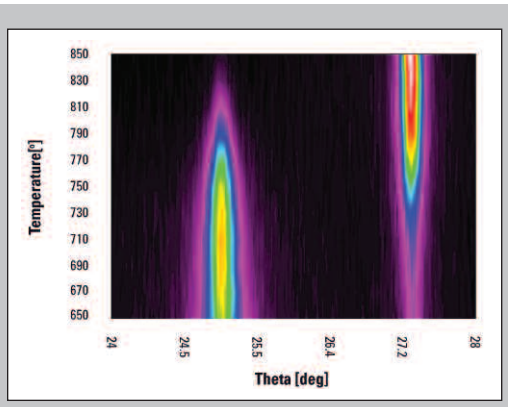
Phase identification by the search-match method using the ICDD database



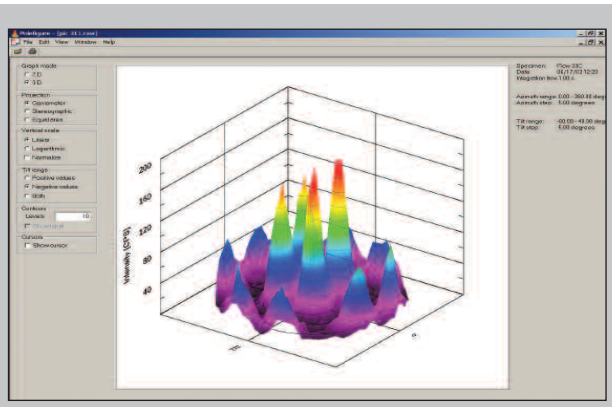




1. High temperature chamber with sample spinner (Anton Paar HTK 1200)
2. Parabolic mirror and thin film collimator
3. Capillary stage for small and/or reactive samples
4. 12 position sample changer for automated batch processing
5. Pinhole collimator for point focus for small samples
6. High temperature chamber for thermal property analysis (Anton Paar HTK 16/20)
7. Environmental chamber for reactive samples analysis
8. High/Low temperature chamber with liquid nitrogen Dewar (Anton Paar TTK 450)
9. Pressure and temperature reactor chamber with sample spinner (Anton Paar XRK 900)



Temperature transition diagram of TiO2 polymorphic phases: Anatase and Rutile



3D Polefigure of an NBT film

Technical specifications for the ARL X'TRA

Electrical requirements	Single phase 200-250V, 32A, 50-60 Hz
Shipping weight	600 kilograms, including packaging.
Water requirements	4.0 liters per minute, 4 - 6 bars, 16°-24° C
Temperature:	18° to 32° C
Filter	Five-micron filter system is recommended to remove particles from water supply.

Dimensions of the instrument
(Red: dimensions for shipment)

Laboratory Solutions Backed by Worldwide Service and Support

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Australia
+61 2 8844 9500 • analyze.au@thermo.com
Austria
+43 1 333 50340 • analyze.at@thermo.com
Belgium
+32 2 482 30 30 • analyze.be@thermo.com
Canada
+1 800 532 4752 • analyze.ca@thermo.com
China
+86 10 5850 3588 • analyze.cn@thermo.com
Denmark
+45 70 23 62 60 • analyze.dk@thermo.com
France
+33 1 60 92 48 00 • analyze.fr@thermo.com
Germany
+49 6103 408 1014 • analyze.de@thermo.com
India
+91 22 6742 9434 • analyze.in@thermo.com
Italy
+39 02 950 591 • analyze.it@thermo.com
Japan
+81 45 453 9100 • analyze.jp@thermo.com
Latin America
+1 608 276 5659 • analyze.la@thermo.com
Netherlands
+31 76 587 98 88 • analyze.nl@thermo.com
South Africa
+27 11 570 1840 • analyze.sa@thermo.com
Spain
+34 91 657 4930 • analyze.es@thermo.com
Sweden / Norway / Finland
+46 8 556 468 00 • analyze.se@thermo.com
Switzerland
+41 21 694 71 11 • analyze.ch@thermo.com
UK
+44 1442 233555 • analyze.uk@thermo.com
USA
+1 800 532 4752 • analyze.us@thermo.com

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