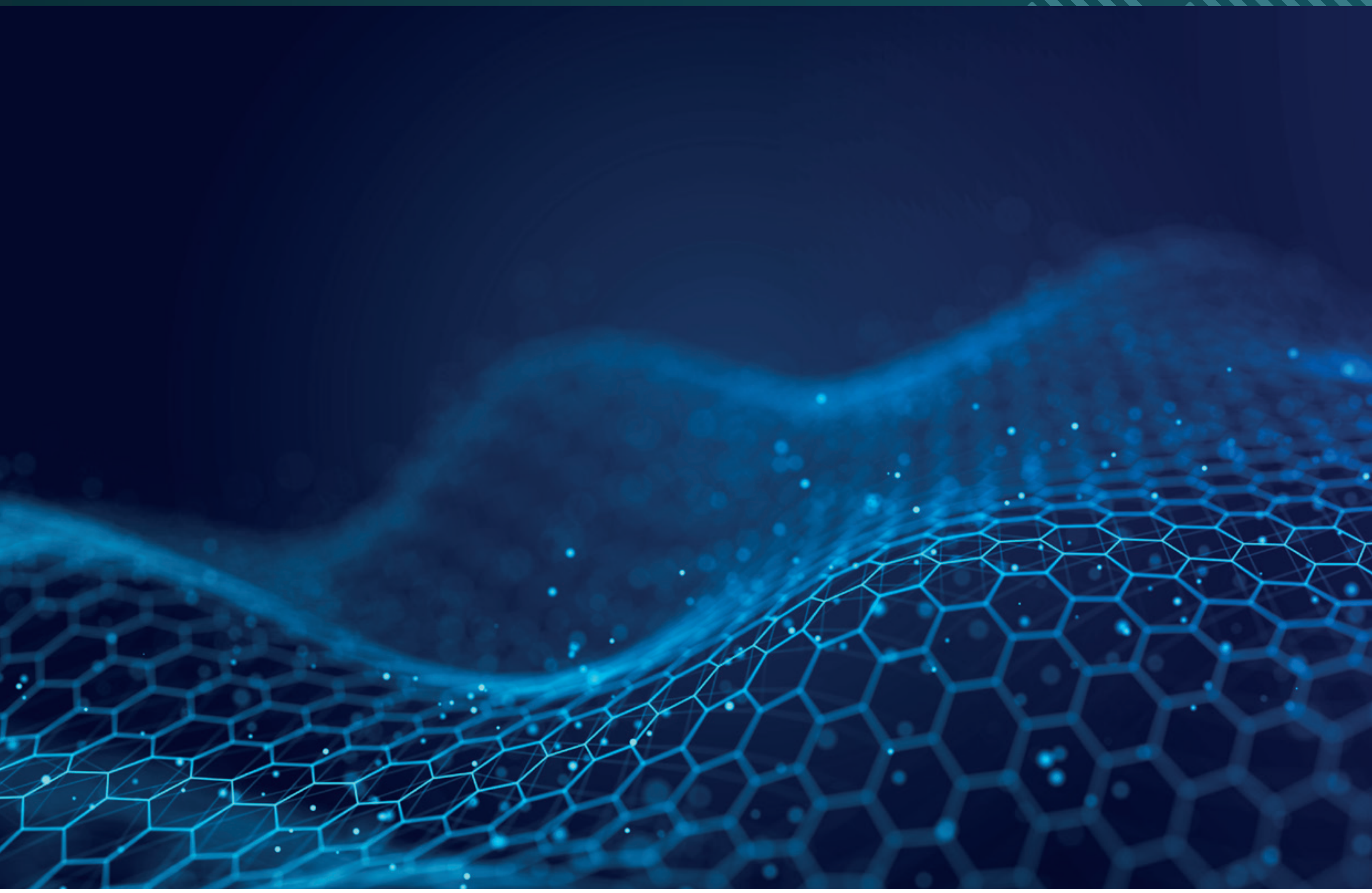
Analytical technologies for catalyst manufacture and research

Understand the physical properties and chemical constitution of your catalyst materials



The power of heterogeneous catalysis

Heterogeneous catalysts have been at the heart of the chemicals industry for over 100 years, enabling transformations ranging from the production of commodity and fine chemicals to the removal of harmful gases from vehicle exhaust.

But now, we're witnessing the emergence of a new era in catalyst application, driven by the demand to extract value from waste feedstocks, find environmentally benign routes to transport fuels, and use scarce elements more wisely.

Staying ahead in this race to capitalize on emerging markets requires a better understanding of the underlying physical and chemical properties of catalyst materials – and that's where Malvern Panalytical can help. With over 60 years' experience in designing, manufacturing and supplying analytical instruments for all types of materials, we're ready to advise you on investigating the material properties of your heterogeneous catalysts, whether you're in research or production.

Alongside their long-established roles in synthesis of industrial chemicals, polymers and petrochemical-derived fuels, catalysts are increasingly playing a central role in the green transition to circular industrial processes with a low environmental footprint. Malvern Panalytical's equipment can help you achieve your sustainability goals by understanding the material properties of catalysts in more depth than ever before.

State-of-the-art equipment for material characterization

Carrying out precise chemical transformations mediated by heterogeneous catalysts requires materials that are highly complex, usually in terms of their physical microstructure, and often in terms of their chemical composition as well.

Characterizing these materials in depth is of course an inherent part of the research and development of catalysts, but it's also valuable during catalyst manufacture, especially for monitoring product quality and consistency, and for troubleshooting issues arising during production or the end application.

At Malvern Panalytical, we provide a range of instruments that can help you investigate and monitor the physical, chemical and microstructural properties of your catalyst materials at every stage of research, development and manufacture. These include systems for:

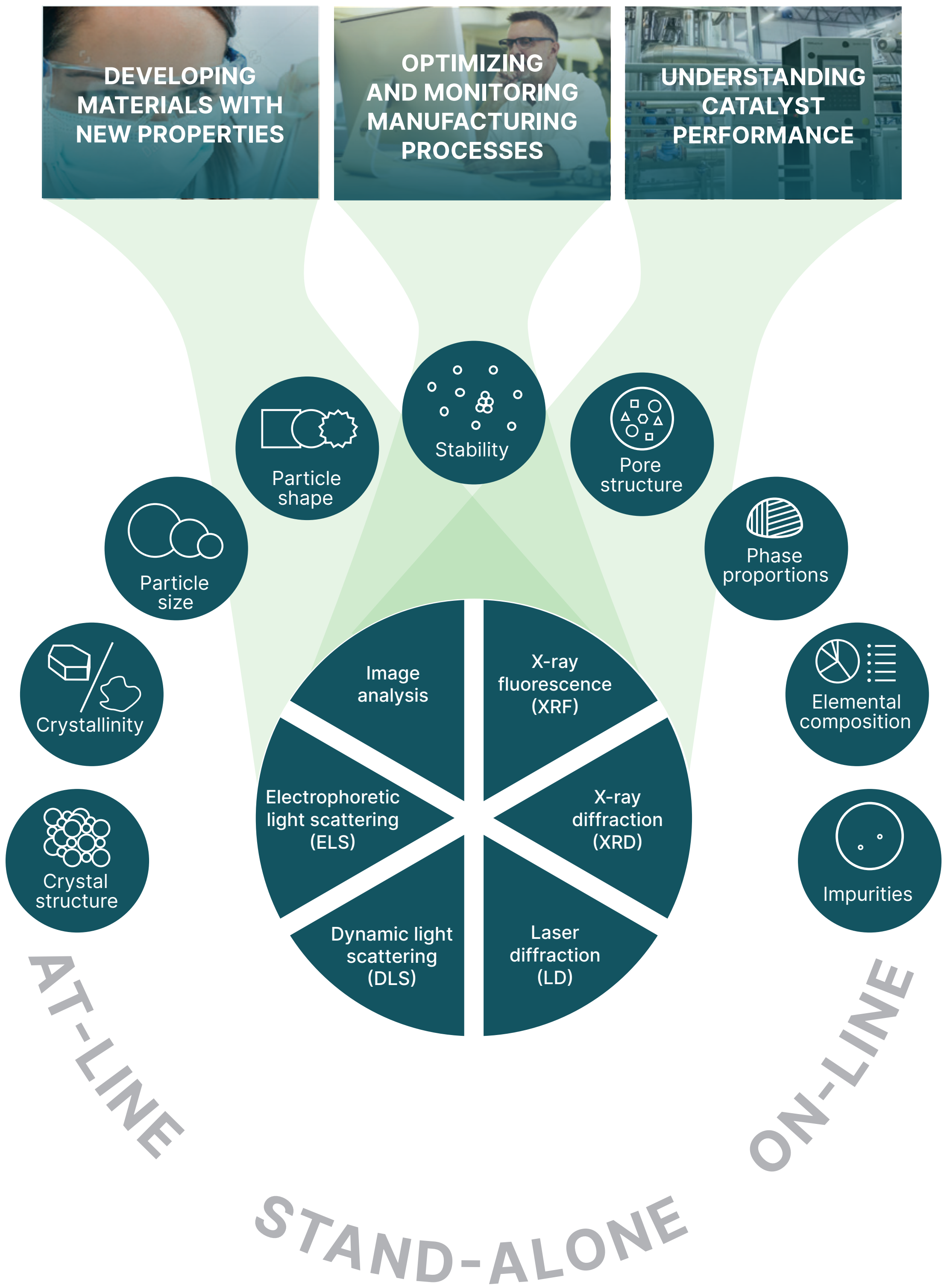
- **Stand-alone analysis** – for all classes of off-line measurement from sophisticated *operando* research through to processing. As well as straightforward analysis of individual samples, most systems have sample changer options that help you to create batch measurements and streamline your routine analyses.
- **At-line analysis** – for enabling robot automation of sample handling, measurements, and analyses. Many samples can be collected during a processing cycle and rapidly analyzed with minimal user intervention, freeing your analysts to deal with the troubleshooting.
- **On-line analysis** – for non-destructive measurement of materials during processing. These systems allow real-time analyses of materials flowing through production lines, making them ideal for quality control.

So whether you're researching new catalysts to meet emerging challenges, or simply ensuring that your products maintain their reputation as the best in the field, we're in an excellent position to support you with analytical equipment and expert advice.

Matching analytical solutions to industry challenges

Malvern Panalytical's products allow measurement of a variety of material properties, providing solutions for the three main challenges faced by catalyst manufacturers and researchers.

Pages 5–7 describe these three challenges in more detail, followed on pages 8–15 by details of the analytical methods, and the equipment options available for each of them.

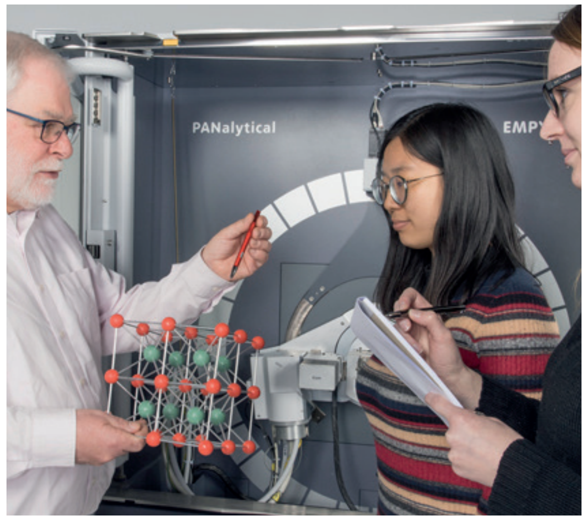


Developing materials with new properties

Developing new catalysts for the rapidly changing demands of society is a difficult balancing act, requiring the fine-tuning of catalytic activity and/or selectivity, alongside a deep understanding of surface properties. This is all set against the scarcity and cost of many catalytically active elements, and the need to improve sustainability and lessen environmental impact.

Opportunities to decarbonize our economy present new challenges. For example, electrolytic processes that intend to use renewable sources of energy such as wind and solar power must be able to cope with fluctuations in electricity supply, which requires a fundamental shift in catalyst design away from the steady-state paradigm that the catalyst industry has long followed.

Fortunately, our analytical methodologies can help both academia and industry to resolve these challenges.



- **Reducing reliance upon scarce elements**
  Platinum-group elements underpin the performance of many catalytic systems, but they are expensive, and upscaling catalytic processes is limited by their availability. Therefore, as well as developing new catalysts using more readily available elements, there is a need to minimize loadings of these elements, by complete or partial substitution. Our Zetium and Epsilon XRF instruments address both challenges, by enabling researchers to measure elemental concentrations in new catalytic materials.
- **Understanding hierarchical pore size distributions**
  The micro-, meso- and nano-pores often needed in catalyst materials can be highly ordered and present at multiple length scales, providing a complex system of surfaces and hence a much greater catalytic activity than a non-porous material. In addition to XRD structure refinement, X-ray scattering methods such as small-angle X-ray scattering and reflectometry are available on our Empyrean XRD systems. Together these can probe porosity scaling from lattice voids to nano-pores.
- **Understanding local structure**
  The local atomic structure of catalytic materials is central to their activity, but can change during reaction processes. Solving the structures of strongly crystalline materials can be carried out on our Aeris and Empyrean XRD systems, but the Empyrean can also be used to investigate subtle changes in amorphous or highly defective structures using total scattering and pair distribution function analysis (PDF). This avoids the need to resort to synchrotron sources, and so enables more flexible, timely analysis.
- **Characterizing substrate particles during loading**
  Characterizing substrate particles before and after loading with catalytically active material may be useful to ensure that no undesirable changes in particle morphology or chemistry have taken place. As well as our Mastersizer and Insitec LD and Zetasizer DLS instruments for characterizing particle size, we also offer Aeris and Empyrean XRD systems, for obtaining a detailed understanding of phase changes.
- **Controlling the distribution of active sites on catalyst supports**
  Impregnation of active metals onto a catalyst support is one of the major routes to heterogeneous catalysts, but an uneven distribution of metal nanoparticles can give rise to poor levels of activity. This distribution is influenced by the surface charge of the catalyst support, making measurement of zeta potential by ELS useful during the development phase of catalyst research.

Optimizing and monitoring manufacturing processes

Good process control is central to catalyst manufacturing, and can be used to check that materials at all stages of production meet the desired standards, and also to optimize processes (especially on scale-up) to reduce costs. From an analytical standpoint, part of the challenge is the timeliness of a measurement, which is where equipment capable of making at-line measurements really comes in to play, by helping to streamline decision-making, and so making for a clear return on investment.

And in addition to all this, equipment for industry needs to be easy to use – so it's reassuring to know that a priority for us is ensuring that it is easy to generate sound datasets and robust insights.

- **Using raw materials responsibly and sustainably**
  The scarcity and price of Pt-group metals, and the desire to source catalytic materials from sustainable, ethical sources, is placing pressure on manufacturers to maximize recoveries to reduce costs. Our Zetium and Epsilon XRF instruments can assist by providing accurate information on the abundance of high-value elements in materials at all stages of catalyst manufacture and recycling, helping you ensure that you're making the best use of valuable materials.
- **Checking that purity specifications have been met**
  Assessing the purity of raw materials or finished catalysts – or keeping an eye on composition during manufacture – requires methods for determining composition that are both quick and reliable. Our Zetium and Epsilon XRF systems provide an easy-to-learn and hassle-free way of assessing elemental composition. Where more detail is needed, experiments using our Empyrean and Aeris XRD instrument can provide additional information on phase purity and (for example) amorphous-to-crystalline ratios.
- **Controlling particle size and shape at all stages of manufacture**
  Particle size and shape distributions are fundamental metrics of all heterogeneous catalysts, and monitoring these is vital at all stages of manufacture, particularly to understand the results of processing stages such as powder mixing, nucleation, precipitation, granulation, spray drying, calcination, milling, extrusion or tableting. Our Mastersizer LD system offers trouble-free monitoring of particle size at the micro and nano- scale (and it's also available as an on-line format, Insitec), while for nanoparticles, DLS analysis with Zetasizer is an alternative option. Automated image analysis with Morphologi (or our on-line Hydro Insight system) additionally can spot shape anomalies that can be a good predictor of particle flow problems.
- **Predicting washcoat stability**
  The stability of liquid dispersion washcoats is partly determined by the surface charge of the particles, with a high surface charge generally preventing aggregation. The zeta potential of a surface is a good indication of surface charge, so measuring it using our Zetasizer ELS instruments, and combining it with knowledge of rheological properties, can be used to optimize dispersion conditions for a given suspension. The result? Maximum washcoat stability, and a final product with fewer defects.
- **Troubleshooting during manufacture**
  Even the most well-controlled catalyst manufacturing processes will occasionally run into difficulties. When that happens, you need to be able to call upon a range of techniques to investigate whether there is unwanted aggregation, attrition or sedimentation, or an undesirable change in phase or surface chemistry. Malvern Panalytical's breadth of instrumentation compatible with on-line or at-line analysis is your ally when resolving a production issue. You might want LD (Mastersizer or Insitec) to check on particle size anomalies, XRF (Zetium, or Epsilon Xflow or Xline) to investigate deviations in stoichiometry, image analysis (Morphologi or Hydro Insight) to investigate powder-flow difficulties, or XRD (Aeris) to resolve suspected problems with structure and composition.

Understanding catalyst performance

The performance of a catalyst is dictated by more than just what happens in the reactor initially – how its performance changes over time is a key factor in its commercial success. Early-stage research may not always give the answers you need, because the performance of small quantities of well-defined catalytic materials under ambient conditions rarely translates seamlessly to larger-scale conversions under higher temperatures and pressures.

Therefore, *in situ* analysis of catalysts at industrially-relevant scales over longer time-frames is a fruitful line of enquiry for both academia and industry, and one in which Malvern Panalytical's equipment can play a role. A further benefit of our XRF and LD systems is that their off-line and on-line versions use the same technology – aiding transfer of results from early-stage research to late-stage development.

- **Scaling-up research methods**
  A major challenge for catalyst researchers in academia and industry is scaling up lab methods to the pilot and production scales. Processes that have worked well during the research phase may suffer unexpected problems when scaled-up; and formulating catalysts to be mechanically stable when pelletized is also a common challenge. Two on-line instruments offered by Malvern Panalytical – the Epsilon (for XRF) and Insitec (for LD) – are perfect for incorporating into scaled-up production processes, and come complete with software to automate measurement and analysis.
- **Studying catalyst physical stability and lifetime**
  Under the elevated conditions of pressure or temperature used in a reactor, the solvation characteristics or friability of a catalyst may be quite different from that studied under less extreme conditions during the research stage. Understanding the mechanical stability of a catalyst is also a factor when designing the pelletization process. The extent to which a catalyst mechanically breaks down during use can be investigated by particle size analysis on our Mastersizer or Insitec LD instruments, while particle image analysis using our Morphologi equipment can provide information on particle shape, aiding understanding of breakdown mechanisms.
- **Looking at catalysts *operando***
  Initial research into catalyst systems often involves a mechanistic understanding at the atomic and molecular level. However, when applied in real-world scenarios, the catalytic environment may be considerably more complex. For example, to reduce reactor downtime, a catalyst may be required to operate under gradually changing physical and thermal conditions. Identifying the range of acceptable reaction parameters requires instruments that can report on key metrics *in situ* over time – such as our on-line Epsilon Xflow system for elemental composition, or our on-line Insitec LD instrument for particle size. In addition, you can perform *operando* phase- or structural analysis measurements under simulated reactor conditions on our Empyrean XRD platform.
- **Investigating recyclability**
  When the catalytically active metals are expensive or in short supply, minimizing losses at every stage of the product lifecycle can have a big impact upon profit margins. To that end, many manufacturers are investigating options for recycling spent catalysts, or extracting valuable metal residues at other stages of the production process. Understanding the matrix in which valuable elements are present – and their levels – is a natural application of abundance analysis using XRF, and our Zetium and Epsilon instruments are ideal for this purpose. The latter also has two modules (Xflow and Xline) that allow on-line monitoring of elemental composition in liquids and coatings, respectively.

Easy-to-perform elemental analysis

Quickly obtaining data on elemental composition using X-ray fluorescence spectroscopy

Deviations in the chemical composition of catalysts can have a significant impact upon final performance, making analysis of elemental composition an indispensable part of the manufacturing process.

X-ray fluorescence spectroscopy (XRF) is a widely-used technique for routine screening or quick analysis of elemental composition, which does away with the inconvenience of sample digestion. It is compatible with loose powders, pressed pellets, fused beads, linished metals or liquids, and is also a proven method for the mainstream assaying of precious metals.

Our XRF spectrometers, the Zetium and Epsilon ranges, provide an easy-to-learn way to analyze elemental composition, monitor trace elements and detect impurities. And they're cost-effective too, with the cost per analysis being up to three times lower than with ICP. Our XRF systems also excel in terms of sensitivity, with measurements of matrix element compositions and ppm-level impurities being more reliable than with ICP.

Zetium, by combining the best of wavelength-dispersive and energy-dispersive systems, offers maximum sensitivity (down to 0.1 ppm), and is suitable for stand-alone R&D applications and process control across the full elemental range.

The system is stable over periods of months, meaning that measurements don't drift, and rendering time-consuming re-standardizations unnecessary. Carrying out XRF measurements is also far quicker than fire assays.

Using Zetium, you'll be able to:

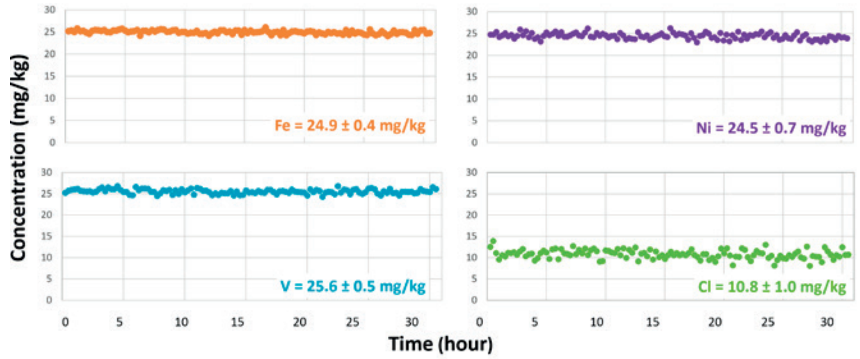
- Measure all elements from Be to Am, including alloys and metal oxides
- Quantify valuable elements such as Pt, Rh and Pd in high-grade alloys
- Detect low concentrations of catalyst poisons such as Cl, S, Sn and Pb
- Quickly determine metal ratios in catalytic supports (such as Si/Al ratio in zeolites)
- Carry out spot-mapping of small areas
- Combine with XRD for insights into phase identity.

Epsilon 4 and **Epsilon 1** are compact and easy-to-maintain energy-dispersive systems that offer stand-alone and at-line solutions for analysis of solids and liquids. Their sensitivity can extend down to 1 ppm and beyond, making them ideal when you need to make a measurement during a process.

Two further Epsilon 1 instruments extend its applicability to continuous (on-line) measurements – the **Epsilon Xflow** for liquids, and the **Epsilon Xline** for roll-to-roll coatings.

Using the Epsilon systems you'll be able to:

- Measure all elements from F to Am, including alloys and metal oxides
- Obtain direct insights into production processes using at-line elemental analysis with sample changer and automation options
- Continuously monitor deviations in stoichiometry of slurries, as an alternative to downstream analysis of the resultant solid materials (Epsilon Xflow)
- Continuously monitor reactor contents on-line (Epsilon Xflow)
- Continuously measure elemental composition during roll-to-roll deposition processes (Epsilon Xline).



On-line XRF analysis using the Epsilon Xflow. Here, the abundance of four catalyst inhibitors in a fluid catalytic cracking process was tracked over a period of 30 hours.

Sample preparation for XRF and ICP

XRF uses fusion to create glassy sample glass discs which (unlike analysis of powders or pressed pellets) eliminate any effects of particle size, mineralogy and surface roughness. Catalyst materials are particularly amenable to fusion, and give highly reproducible results with wavelength-dispersive XRF systems. The same sample preparation equipment can also be used to make the peroxide or borate solutions that are needed for ICP analysis.

Whether you're using XRF or ICP, we can provide equipment that will ensure effortless sample preparation. The **Claisse LeNeo** is an easy-to-use single-position instrument that is ideal for occasional or low-throughput use. The new **FORJ** system, on the other hand, with capacity for up to 12 samples, is better-suited to high-throughput scenarios. It's also the world's fastest, safest and most accurate fusion instrument for sample preparation, designed for minimal maintenance and repeatability of results.

Did you know? We also provide certified reference materials (CRMs), together with the expertise you need to set up bespoke calibrations and standard operating procedures.

High-resolution particle size distributions

Obtain new insights with precision analytical instrumentation

Optimizing the particle size distribution is central to several stages of catalyst production, from the preparation of precipitates, powders and washcoats to the molding of the final catalyst block.

To meet this diversity of needs, two easy-to-use instruments are offered by Malvern Panalytical for measuring particle size – the Mastersizer 3000 and the on-line Insitec system. Both employ the technique of laser diffraction, which unlike grindometers provides information on the overall size distribution of catalyst particles, not just the largest particles present.

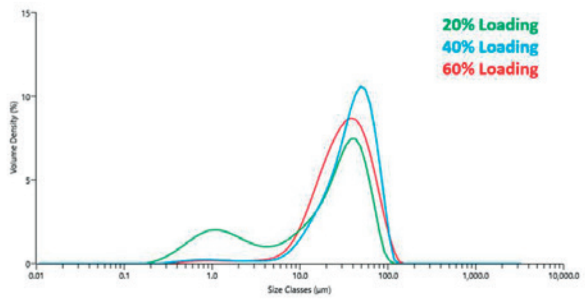
In addition, because the data represents the properties of the whole material, the results are statistically superior to microscopy (which depends upon selecting a representative sample). Laser diffraction is also high-resolution, easy to perform and operator-independent, making it useful to replace or complement other methods (for example, those used for benchmarking or calibration).

The **Mastersizer 3000** delivers rapid, accurate particle size distributions for powders, suspensions and emulsions over the size range of 0.01–3500 µm – making it a dependable and versatile instrument for the majority of particle sizing applications.

And with a small footprint, plus effective sample preparation capabilities, flexible reporting, and options for process automation, Mastersizer 3000 is guaranteed to be a success in the research lab and on the production line.

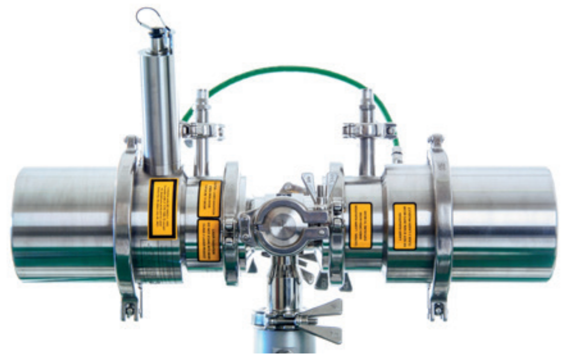
Using Mastersizer 3000, you'll be able to:

- Quickly and easily obtain high-quality, reliable data about particle size
- Check the size of precipitates and post-milled powders, particularly to avoid over-milling
- Estimate the specific surface area (SSA) of catalyst supports (note that values tend to be smaller than obtained with BET measurements, which are sensitive to the internal pore structure)
- Control particle size in washcoats (whatever the solvent system), and so optimize their impregnation into (or adhesion onto) catalyst supports
- Optimize the rheology and compressibility of the catalytic powder at the final shaping stage
- Use pressure titrations to understand the risk of catalyst attrition in fluidized-bed reactors.



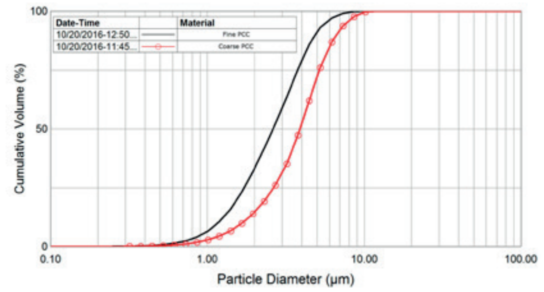
Using Mastersizer 3000, size distributions for three Pt loading levels on Vulcan XC-72 carbon black were accurately generated, showing a bimodal distribution for the lowest loading.

The **Insitec** system is tailored to on-line monitoring, providing real-time analysis every few seconds for powders, emulsions, sprays and slurries over a specified size range (lenses are available for bands covering the size range 0.1–2500 µm). This makes it ideal for fast-moving production environments, facilitating quick remedial action, and so helping to reduce waste and make workflows more efficient.



Using Insitec, you'll be able to do many of the measurements you can do on the Mastersizer, plus:

- Obtain direct insights into production processes using on-line size analysis (and you can use Insitec alongside Epsilon Xflow or Epsilon Xline for on-line elemental abundance information)
- Troubleshoot suspected particle size issues during milling
- Check particle size evolution over time during sedimentation.



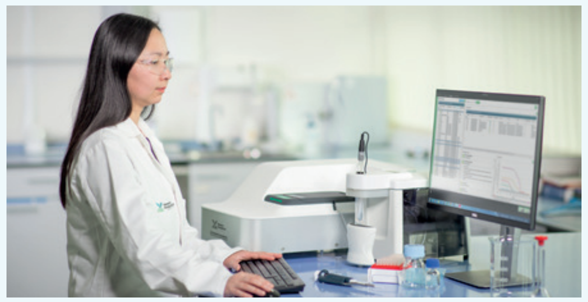
As the process requirements are changed and the manufacturing feed lines are adjusted, successive measurements in a 1 hour period track the change from coarse (red) to fine (black) precipitated calcium carbonate powder.

Understanding nanoparticle distributions using dynamic light scattering

Laser diffraction is ideal for the vast majority of catalyst applications, but when the particles being researched are below 600–800 nm (as, for example, in some highly-dispersed colloids), dynamic light scattering (DLS) offers greater sensitivity, and below 200–300 nm becomes the recommended option.

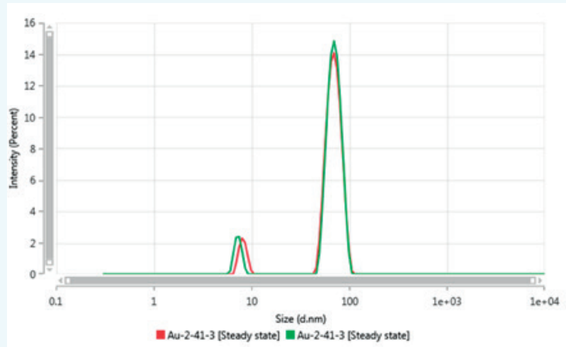
Moreover, because DLS systems are far less expensive and more user-friendly than transmission electron microscopy, they are ideal for researchers needing to routinely and quickly measure size distributions in this range.

The **Zetasizer Advance** range are the most widely used instruments for measuring both particle size and zeta-potential. Ask about our MADLS® technology and how it uses advanced algorithms to combine multi-angle data for greater sensitivity and precision!



Using Zetasizer Advance systems, you'll be able to:

- Quickly and easily obtain high-quality, reliable data about nanoparticle size
- Control nanoparticle precipitation – for example, of precious metal nanoparticles in a washcoat
- Maximize the shelf-life and efficacy of washcoats for nanoparticle absorption onto substrates
- Predict issues with particle agglomeration or settling before they happen.



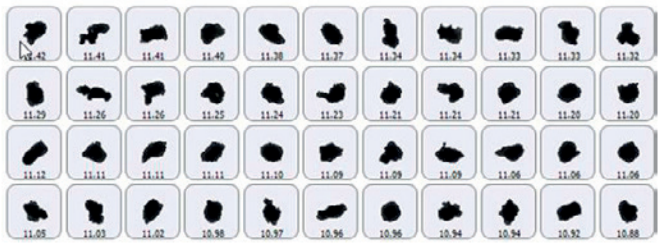
Intensity size distribution as measured by MADLS® of gold nanoparticles. Further measurement using polarized light revealed that the smaller peak arose from rotation of some elongated particles.

Statistically-sound particle shape analysis

Acquiring detailed particle morphology data using image analysis

Particle shape is a complex topic in catalyst manufacture, but one that is important to understand because of its numerous effects on processing and (ultimately) catalytic activity.

The manual microscopy methods historically used to analyze shape were highly time-consuming, and have now been superseded by automated image analysis, for example using our Morphologi 4 and Hydro Insight instruments. This automation also means that analysis can be performed on large sample sets – typically 10,000 to 500,000 particles.



Partial catalog of a 40% Pt/C catalyst powder used in a catalytic ink imaged by Morphologi 4. Although the agglomerates are roughly spherical, their roughness could affect the ease of dispersion in the ink and the formation of porosity during deposition and drying.

Morphologi 4 uses automated static image analysis to provide data on over 20 morphological particle parameters, for dry powders, wet suspensions and particulates collected on filters, in the range 0.5 to >1300 µm. Moreover, SOP control and software filters ensure robust, repeatable measurements, even for low-contrast particles, or where small fragments, image anomalies or touching particles are present.

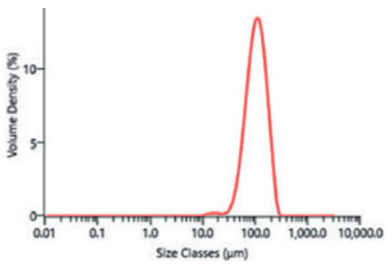
Using Morphologi 4, you'll be able to:

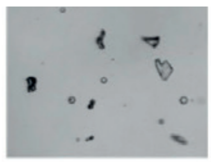
- Obtain highly detailed, statistically robust particle shape and size distributions
- Obtain information sphericity/irregularity or the presence of anomalous particles
- Study the microstructural properties of carbon materials (which are often used as metal-free supports).

Hydro Insight is a dynamic imaging accessory for wet dispersions that sits alongside the Mastersizer 3000 (see page 10), and which can image suspended particles at up to 127 frames per second, and so provide real-time information on particle shape.

Using Hydro Insight, you'll be able to:

- Acquire on-the-spot information on particle shape in wet dispersions
- Respond rapidly to an imperfect batch
- Troubleshoot issues arising during method development.





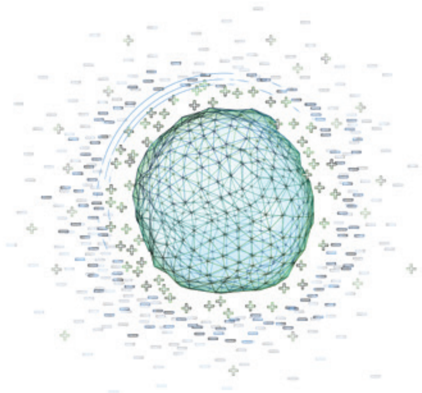
With Hydro insight, powerful image processing software analyses successive high resolution frames, presenting histograms of parameters, such as the size class histogram shown here.

Reliable zeta potential measurements

Unpacking the role of surface charge using electrophoretic light scattering

In catalyst manufacture, the charge on particles has an important role to play in adhesion and aggregation processes, and this propensity to develop a charge can be quantified by the zeta potential using electrophoretic light scattering (ELS).

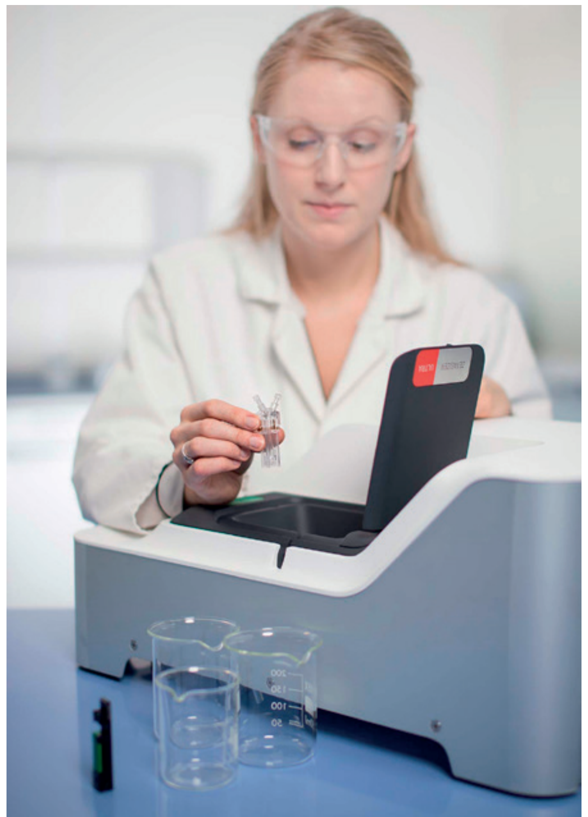
To enable catalyst manufacturers to accurately measure zeta potential, we offer the Zetasizer Advance range of instruments – the successor to the highly successful and market-leading Zetasizer Nano.



The **Zetasizer Advance** range of instruments is ideal for measuring zeta potential with excellent accuracy, repeatability and consistency in process or laboratory environments.

When highly conducting samples are being studied, Zetasizer has you covered, with 'constant-current zeta' technology keeping the current unchanged even when ions build up on the electrodes, and the mixed-mode 'M3-PALS' method avoiding distorted results caused by electro-osmosis.

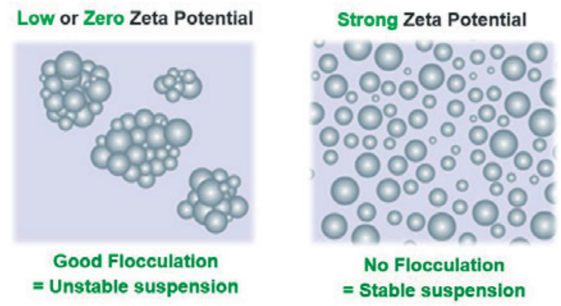
Because all colloidal samples are different, the



Zetasizer's multipurpose titrator **MPT-3** allows you to set up titrations in different ways to suit your sample and find the optimum pH for stability.

Using Zetasizer Advance, you'll be able to:

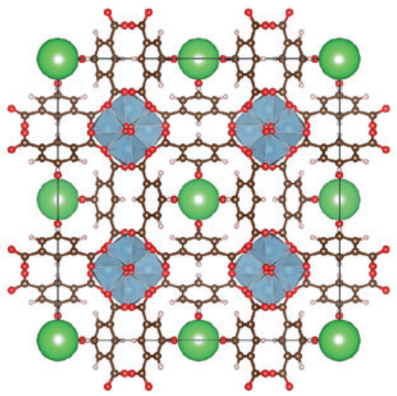
- Find out everything you need to know about zeta potential of your particle dispersion
- Understand the root causes of unstable particle dispersions or poor particle adhesion
- Optimize aggregation of catalytic powders, to aid compressibility for tableting/extrusion
- Optimize formulation stability by titration against pH or concentration of steric dispersant
- Ensure optimized distribution of active sites following active-material impregnation.



The formation of large particles due to aggregation and flocculation is enhanced when the net charge of the particles is close to zero. The pH of a dispersion can be changed to prevent floculation and achieve stability.

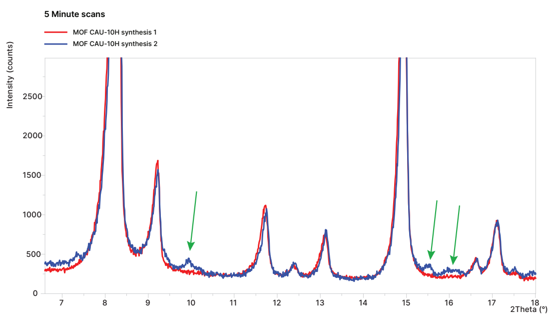
Powerful phase analysis

Investigating catalyst phases in unmatched detail using X-ray diffraction



Understanding the atomic structure of crystalline phases is an essential part of determining the structure–performance relationships and reaction mechanisms of heterogeneous catalysts, and X-ray diffraction (XRD) has long been valued by materials scientists for this purpose.

Two XRD instruments from Malvern Panalytical are valued by those in the catalyst industry for the analysis of catalyst materials: Aeris and Empyrean.



To refine your processing parameters, you need to analyze them quickly. With Aeris, even five-minute scans can immediately show whether a processing parameter is affecting crystalline phase purity. This figure shows two five-minute measurements of two metal-organic framework (MOF) samples in reflection mode, each processed under different conditions. Using Aeris, you can rapidly optimize these processing parameters for the best quality material – every time.

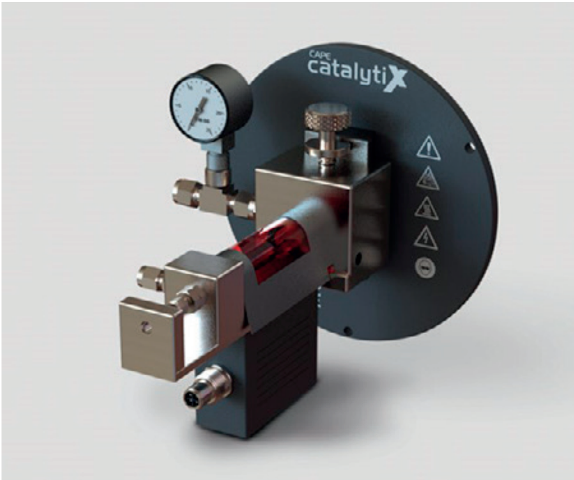
Ask us about the new *in-situ* catalyst cells!

Aeris is a compact diffractometer offering industry-ready measurement and data analysis on a variety of powder diffraction configurations.

Using Aeris doesn't require expertise in XRD. And with the option of belt automation, sample processing is easily scaled up, with a typical sample taking just a few minutes – helping you improve production efficiency, reduce costs and control the quality of the finished product. In addition, expertise packages can be employed to set up fully automated phase composition results for your routine samples, so that you can streamline your QC and trend analysis.

Using Aeris, you'll be able to:

- Automate the routine quantification of phases (e.g. γ-alumina, zeolites)
- Measure the degree of crystallinity of active phases
- Determine the point of termination of the crystallization step during manufacture
- Combine phase information with XRF data for insights into element distribution
- Carry out grazing-incidence measurements to improve data quality for surface layers and coatings
- Carry out transmission XRD measurements at low angles, to study microporous materials.

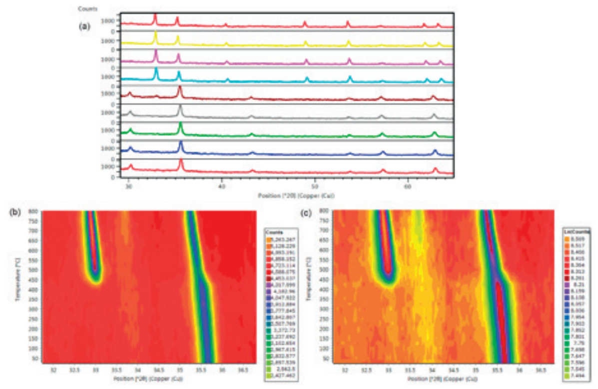


The **Empyrean** range of instruments are versatile, sophisticated platforms that offer the widest possible choice of measurements, meaning you're literally ready for anything.

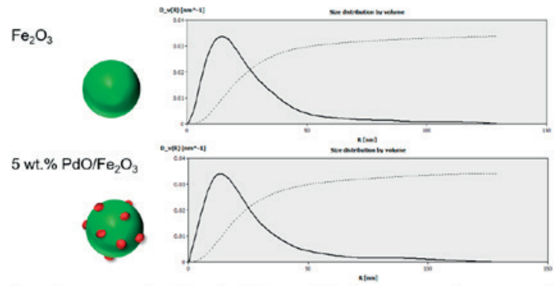
Ideally suited to R&D laboratories, Empyrean is the only XRD system with the ability to measure all sample types on a single instrument. And with automated MultiCore optics, switching between measurement or sample types is easier than ever before.

Using Empyrean, you'll be able to do everything you can with Aeris, plus:

- Have access to the widest possible range of XRD experiments, for unparalleled flexibility
- Carry out extensive *operando* or *in-situ* measurements on catalytic materials, for real-world insights
- Determine the larger dimensions (and pore sizes) of catalyst scaffolds using small-angle X-ray scattering (SAXS)
- Understand the local structure of amorphous materials using X-ray total scattering and pair distribution function (PDF) analysis.

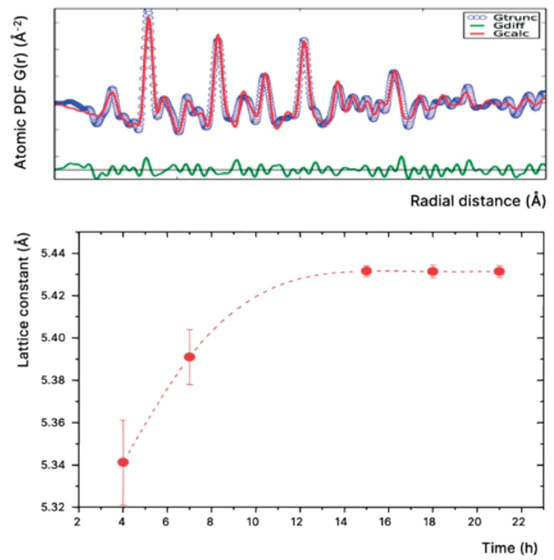


In-situ monitoring of the thermal response of catalyst material Fe_2O_3 and PdO/Fe_2O_3 between 250°C and 800°C using an Anton Paar HTK 1200N stage on the Empyrean platform. **(a)** A stack plot of XRD data files shows a phase transition from maghemite to hematite. **(b)** An isolines plot for the two Fe_2O_3 phases indicates this phase transition takes place at around 450°C. **(c)** An isolines plot for PdO/Fe_2O_3 shows a sharpening of the primary peak corresponding to PdO at approximately 550°C, indicating an increase in the average crystallite size of PdO and loss of the small surface domains on the support material.



Chemical composition	SSA [m^2g^{-1}]	SSA from BET [m^2g^{-1}]	R50 [nm]	R_{TEM} [nm]
Fe_2O_3	38.8	37.3	21.0	20 – 50
5 wt.% PdO/ Fe_2O_3	40.8	-	20.2	

SAXS measurements of pure Fe_2O_3 and PdO/Fe_2O_3 nanoparticles, carried out on Empyrean, showing an R_{50} value of about 20 nm in both cases, but with a broad size distribution. The derived specific surface area for PdO/Fe_2O_3 is slightly larger than the (BET-validated) value for Fe_2O_3, as would be expected from the presence of active surface sites. Sample courtesy of TU Vienna, Austria.



The X-ray diffraction peaks of nanoparticles can be very broad, preventing the accurate determination of lattice parameters, but models based on pair distribution function (PDF) data, acquired on Empyrean, can be used to derive useful information. **(a)** PDF for CeO_2 nanoparticles; **(b)** the PDF-derived plot of the cubic CeO_2 lattice parameter, shown as a function of synthesis time. The value increased for the first 15 hours, indicating that active particle growth is taking place.

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