



PRESS

ISSUE 2/2018



**A NEW DIMENSION OF
X-RAY DIFFRACTION
ANALYSIS**



**Malvern
Panalytical**
a spectris company

THE POWER OF DIALOGUE



Tanneke Reinders
Vice President Marketing,
Malvern Panalytical

Dear reader,

This edition of our XPress magazine provides me with a great opportunity to introduce myself to you. I joined Malvern Panalytical as Vice President of Marketing on May 1st, and one of my earliest – and most enjoyable – jobs is to contribute to our communication with our customers through this and future Xpress introductions. With a background in the automotive and electronic industries, the world of Malvern Panalytical's technologies is new to me, and I am very excited to explore it. With help from my 2,000 colleagues, I want to gain a deep understanding of our customers' challenges and subsequently help deliver solutions which will solve them. Dialogue with you is critical to achieving this, and I look forward to keeping the communication channels open.

The two new products we introduce within this issue of XPress are great examples of the results of such dialogues with our customers. Both Zetasizer and Emyrean are proven Malvern Panalytical flagship products, which have now been equipped with many new advanced features – features our customers requested when they talked to our specialists about the changing nature of the challenges they were facing, and how they felt our technologies could help. We often hear about the desire for greater efficiency and better ease of use, so our developers have concentrated on answering these requests, resulting in products which revolutionize traditional analytical concepts. The new Emyrean X-ray diffractometer is a good example of this: its new MultiCore Optics provide automatic switching between configurations and increase system utilization by up to 35% because the instrument can run through the night and over weekends without the need for manual intervention.

Generally, communication with our customers has become much easier and

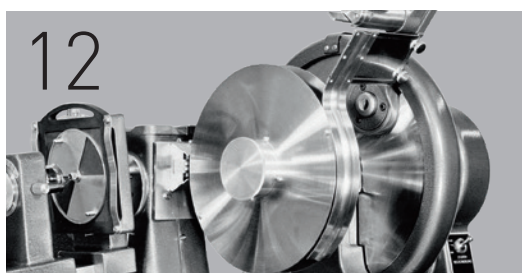
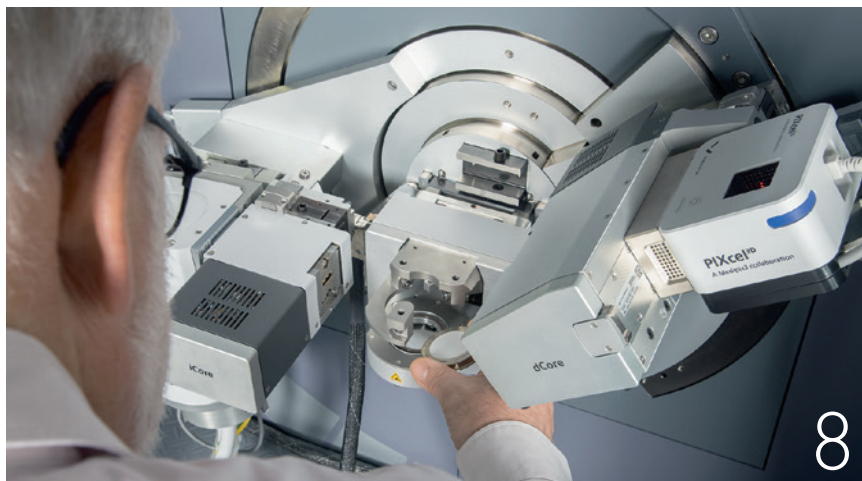
faster with the introduction of digital and social media, so we can keep you up to date on our latest developments through a variety of communication channels. The new General Data Protection Regulation (GDPR) introduced by the European Union on May 25th defines how we must protect the data you provide to us. We have made sure that Malvern Panalytical is fully compliant with these regulations and you can be sure that the required privacy rules will be applied to any data we receive from you. If you were subscribed to our e-mail communications in the past, the new rules require that you opt-in once again to consent to receiving information from us. We, in turn, will make sure that this information is tailored to your needs and interests.

In March, we launched our new website, www.malvernpanalytical.com, which offers you multiple ways of interacting with us. We run a diverse educational series of live webinars where our specialists discuss topics of interest and share their expertise. Additionally, a large library of application notes, whitepapers, articles, customer stories and recorded webinars is freely available to help you delve deeper into a specific application or technology. I find the 'Industries' section especially useful, as it summarizes all solutions we offer for analytical challenges in specific industry sectors – and these are just a few examples! If you want to stay informed about solutions and developments relevant to your business, I can only encourage you to sign up via www.malvernpanalytical.com/register - a one-time action ensuring that you stay in touch with Malvern Panalytical.

I wish you a good and productive summer and hope to talk to you again in the fall. In the meantime don't hesitate to contact us with any feedback via info@malvernpanalytical.com

*With kind regards,
Tanneke Reinders*

IN THIS ISSUE



4 JAN DE NUL **SAVES TIME AND MONEY** WITH MALVERN PANALYTICAL SOLUTIONS

6 **OPTIMIZING** POLYMER SCAFFOLD FABRICATION PROCESSES AT RUTGERS UNIVERSITY

8 ENTER **A NEW DIMENSION** OF X-RAY DIFFRACTION ANALYSIS

10 MEET THE REVOLUTIONARY NEW **ZETASIZER PRO** AND **ZETASIZER ULTRA**

12 THE HISTORY OF **X-RAY TECHNOLOGY**

14 THE NEW MORPHOLOGI 4 RANGE: **IMAGE IS EVERYTHING!**

16 COMBINING FORCES FOR THE **ENVIRONMENT**

18 REVOLUTIONIZING THE **ANALYSIS** OF **BASE METAL SULFIDES**

20 OPTICAL SPECTROSCOPY FOR **INDUSTRIAL AND REMOTE SENSING**

22 **PROTECTING** CUSTOMER DATA

23 PINPOINTING MAJOR CAUSES OF ERRORS IN **XRF ANALYSIS**

PHARMACEUTICAL CHALLENGES AIDED BY A LABORATORY POWDER X-RAY DIFFRACTION SYSTEM



JAN DE NUL SAVES TIME AND MONEY WITH MALVERN PANALYTICAL SOLUTIONS

Jan De Nul is a global market leader in dredging and marine construction activities and executes dredging and land reclamation projects from start to finish: design, development and maintenance of ports, deepening of channels, land reclamation and shore protection works, dredging in the most diverse conditions. Anthony De Vos, Lead Geologist, and Lies De Mol, Engineering Geologist, explain the challenges the company was facing and how they could be solved.

When dredging sand for reclamation, it must be determined whether the sand is appropriate to fill the land and, if not, it must be transferred to a dump zone. This evaluation should preferably be done during transport of the sand; however, previous sieve analyses on site were so time-consuming that it was necessary to use the sand anyway and risk having to remove it at a later stage.

This need for a fast turnaround of samples meant that the company required a particle sizing instrument that could analyze small samples more efficiently, both in laboratories and in the field. Malvern Panalytical's **Mastersizer 3000**, equipped with the **Hydro LV** accessory, was found to be the best overall solution for this challenge. "A big advantage of the Mastersizer 3000 is that it's extremely

compact and easy to transport. The software is also very convenient, meaning you can work with a standard procedure at one push of a button. It's this simplicity and ease which mean that anyone can use the system, with minimal training," says Anthony De Vos.

"Most settings can be pre-installed, reducing the risk of any issues occurring and ensuring reproducibility – this is especially valuable when using the analyzer in an outdoor environment, such as on board a dredging vessel. We have become heavily reliant upon the Mastersizer and purchased a second one in 2017 to guarantee availability in our laboratory. The Mastersizer 3000 with a Hydro LV requires only a few grams per sample, which can easily be transported. This means that we can start analyzing

samples the day we arrive back at the office. Saving time anywhere we can is really valuable to us."

"Malvern Panalytical's technology saves us substantial amounts of time and money."

Anthony De Vos
Lead Geologist at Jan De Nul Group

The company also required an instrument for particle shape characterization that could enable it to estimate the amount of wear on its pipelines during a reclamation project and facilitate cost forecasting. De Vos explains the challenge: "Angular sand particles cause more wear than sand particles that are more circular in shape. The size of the sand particles also has an impact; the bigger the sand particles are, the more impact they will have on the wear. Before a project, we need to estimate the amount of wear as the impact is enormous – reparation costs or new equipment can cost millions. Therefore, accurate particle size and shape analysis is crucial for us."

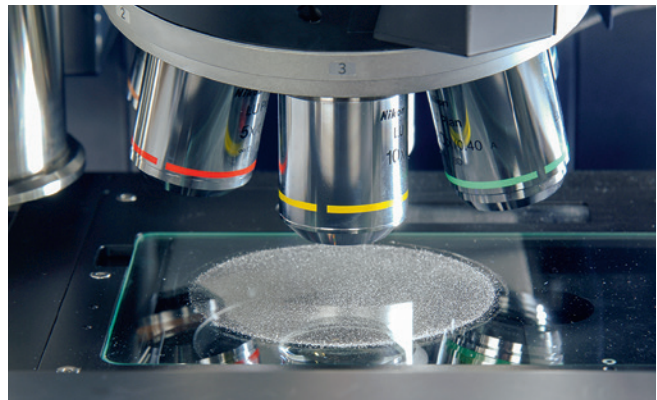


Hydro LV
A large-volume automated dispersion unit that controls wet dispersion of materials for particle size analysis

Mastersizer 3000
A laser diffraction particle size analyzer delivering rapid and reliable particle size distributions for both wet and dry dispersions with easy-to-use, intuitive software with built-in expertise

In summary, De Vos states the impact of Malvern Panalytical's technology to Jan De Nul's operations: "The technology saves us substantial amounts of time and money. Now, we can quickly analyze samples ourselves, reducing the need to outsource analysis. By using these systems on our ships, we can indicate the quality of the sand to our customers before we pump the sand ashore, where time is money."

Following a detailed evaluation of particle morphology analyzers Jan De Nul Group purchased Malvern Panalytical's **Morphologi G3SE**, with De Mol commenting: "We chose the Malvern Panalytical Morphologi G3SE because it delivers the parameters most relevant for us. Another crucial factor was the fact that the instrument enables measurement of particles that are up to several millimeters in size".



Morphologi G3/Morphologi 4
Measures the size and shape of particles from below one micron up to several millimeters in size, using the technique of automated static image analysis.



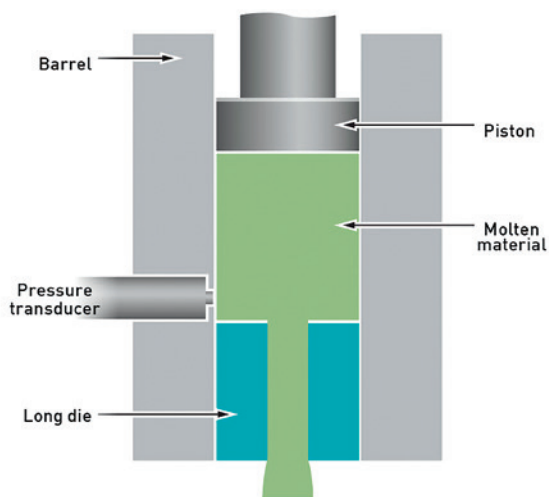
Jan De Nul Group is a leading expert in dredging and marine construction activities, as well as in specialized services for the offshore industry of oil, gas and renewable energy. These core marine activities are further enhanced by Jan De Nul Group's in-house civil and environmental capabilities, offering clients a complete package solution.

The company's professional and innovative solutions are trusted across the industry. Whether it concerns the construction of the new locks in the Panama Canal or a new port complex in Western Australia, together with their customers, they build for further economic development. Jan De Nul Group owes its position as global leader above all to its technical know-how and very diverse fleet. By investing in its own installations, machines and vessels, the Group today has the world's most modern dredging fleet at its disposal.

OPTIMIZING POLYMER SCAFFOLD FABRICATION PROCESSES AT RUTGERS UNIVERSITY

Sanjeeva Murthy, Associate Research Professor at the New Jersey Center for Biomaterials (NJCBM), is a materials scientist with expertise in polymers, biomaterials and biological structures. His current activities at the lab include testing and characterization of new polymers for regenerative medicine, and fabricating them into devices, scaffolds for tissue repair and replacement, biomedical implants, nerve regeneration scaffolds and drug delivery systems. A common denominator in all NJCBM's research projects is the processing of polymers into various forms, fibers, pins and films.

The focus of the NJCBM laboratories is to explore new degradable polymers for a large range of biomedical applications. Before attempting to process their polymers, the most important qualification step is to assess whether the polymer is processable, and if so determine the optimum processing parameters for extrusion and 3D printing with a minimum amount of material. Optimization of the processing parameters is essential to minimize degradation during processing. Temperature, presence of volatiles, duration of processing and shear rate are all factors that contribute to the degradation of the polymer. Previously, NJCBM determined these processing conditions by trial and error on extruders, using up hundreds of grams of polymers.



Capillary rheometry

Based on controlled extrusion of a test material, capillary rheometry enables material flow and deformation properties to be characterized under process-relevant conditions such as high force (or pressure), high shear rate and at elevated temperature.

The sample is extruded through a capillary die, and the resultant pressure is measured at the die entrance. Shear viscosity is calculated from knowledge of the capillary die dimensions, piston speed and pressure.

That's when they realized their need for a capillary rheometer. "Our material is \$20 per gram since it is all custom-synthesized", says Sanjeeva Murthy. "With 5-10 g of polymers we are now able to determine the processing conditions whereas before we needed 200 g on a large extruder. A melt indexer uses the same amount of material but does not provide as much information as the rheometer. Understanding of the shear rate, viscosity, temperature and the pressure required for processing is valuable in our decision to proceed to large-scale operation."

NJCBM needed an instrument that could be used as an extruder, as well as a rheometer, and was easy to use by students with little supervision. They considered both a rotational rheometer and a capillary rheometer but, in the end, they chose Malvern Panalytical's **Rosand capillary rheometer**, which has become a go-to tool for their needs. They chose the small-bore diameter (9.5 mm) rheometer to further reduce the amount of polymer that is necessary to operate the instrument and have modified it to easily extrude filaments.

In a research laboratory, the polymers that need to be processed are often of completely different compositions, and hence their thermal characteristics are unknown. "After initial evaluations using differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA), the first thing we do as soon as we can synthesize approx. 10 g of the polymer, is to try to extrude the polymer," explains Sanjeeva. "If the polymer is extrudable, then while evaluating the rheology of the polymer, we also typically make approx. 100 μm diameter fibers to evaluate the mechanical and degradation behavior of the polymer. This way, the capillary rheometer enables us to screen a large number of polymers and choose the most appropriate one for a desired application." In the evaluation process, they often compare the processability and performance by extruding commonly used polymers such as polylactides.

The utility of the Rosand capillary rheometer in the evaluation of degradable polymers cannot be overemphasized according to Sanjeeva, as these polymers have a tight window for processing. Too low a temperature makes the polymer non-extrudable while too high a temperature causes degradation.

Too short a time in the barrel may not be sufficient to equilibrate the polymer but too long a time might crosslink the polymer. "We use the rheometer to evaluate the additives that might stabilize the polymer during degradation. We also use the rheometer to evaluate the stability of the drugs that are incorporated into the polymer," states Sanjeeva. "We take advantage of the temperature stability of the rheometer to assess the thermal stability of the polymer by keeping the polymer for various lengths of time and at various temperatures, and then measuring their rheology. Such studies are useful in scaling up the extrusion process to large batches of polymers, and also serve as accelerated aging experiments."

One other innovative application for which the group uses rheology is to confirm the molecular weight determinations of a series of polymers of the same composition against their gel permeation chromatography (GPC) method (Malvern Panalytical **Viscotek GPC**). While GPC measures the swollen volume and not the length of the polymer chains, melt viscosity is directly related to the polymer chain length with no interference from solvent and column characteristics present in GPC measurements.

"Even undergraduates are able to produce high-quality data with minimal training. In fact, this was used in connection with the work done by high school students during the summer of 2017 as a part of the New Jersey Governor's School of Engineering &



"Malvern Panalytical's customer service is outstanding."

Sanjeeva Murthy
Associate Research Professor, NJCBM

Technology in a project: 'Fabrication and Characterization of Polymeric Sutures and Brain Stents'."

Malvern Panalytical's reputation for service and technical support was another reason for choosing the Rosand rheometer and Sanjeeva is complimentary about the support

received from Malvern Panalytical to date, commenting that "Malvern Panalytical customer service is outstanding. Their technology consultant is always on call with any help that is required with the instrument. In the future, we plan to use the rheometer for measuring the viscosity of protein solutions and gels."

The New Jersey Center for Biomaterials (NJCBM), a research center of Rutgers, The State University of New Jersey, USA, was founded in 1991 by Dr. Joachim Kohn with the mission to improve patient care and public health through the development and commercialization of future generations of biomaterials.



Degradable stents, drug-eluting hernia patches and antibacterial pacemaker pouches are some of the devices that have resulted from this laboratory and are in clinical use.

Recent successes using the Rosand rheometer:

- Successful extrusion of two tyrosine-derived polymers, one for nerve conduit applications, and the other for meniscus applications
- Extrusion of 4 - 5 mm rods using poly(L-lactic acid) that were machined into bone fixation screws
- Evaluating the suitability of polymers for 3D printing applications and extruding the required diameter (1.75 mm) filaments for use with a fusion deposition modeling 3D printer.



Scaffolds with fibers coated with collagen for meniscus repair



Fibers braided into guidance conduits for nerve regeneration

ENTER A NEW DIMENSION OF X-RAY DIFFRACTION ANALYSIS

Do you want the highest sample throughput in a multi-user, multidisciplinary environment? Would you like to increase the number of students and technicians who can perform advanced analyses and decrease the amount of training that is needed? Do you want to reduce the possibility for mistakes in data collection? Have you ever wondered why X-ray diffractometers are often not used during the night or if they are used, they just repeat the same type of measurement?

With the arrival of Malvern Panalytical's new Empyrean, these issues are now firmly in the past! This successful, well-renowned multipurpose X-ray diffraction (XRD) platform has been installed more than 1000 times worldwide since its introduction in 2010 and has delivered innumerable excellent data sets for all kinds of materials analysis by XRD. With the introduction of the new MultiCore Optics, Empyrean is now taking an evolutionary step towards a new concept for such a multipurpose XRD platform.

Multipurpose X-ray diffractometers are advanced instruments offering functionality for many different applications, each requiring a special instrument configuration. Dedicated optics must be attached manually by an operator with specialist knowledge. Empyrean with new MultiCore Optics – iCore for the incident beam and dCore for the diffracted beam – is changing all that, allowing a variety of measurement types to be run without any manual intervention from an operator.

The new Empyrean can be programmed to run a pre-defined series of measurements during the night or over the weekend. Just enter the task, and Empyrean will deliver the data, potentially with data analysis and reporting included.

This could include screening a batch of many samples or analyzing the various properties of a single sample for a more complete understanding. This automated collection of multiple types of scattering measurements uses your system more efficiently with much less dead time between applications.

The pre-programmed measuring routines considerably simplify training for personnel or students and lower the barrier to perform advanced X-ray applications – just tell the system what you want, and the MultiCore Optics take care of the challenge. As an additional benefit, these standardized routines facilitate the creation of Standard Operating Procedures for example in regulated environments.



COMPREHENSIVE ANALYSIS OF W FILMS ON SI (004)

(Types of films used for RF filters in smartphones)

High-throughput screening in additive manufacturing uses this kind of analytical workflow, which can be entirely automated on the new Empyrean.



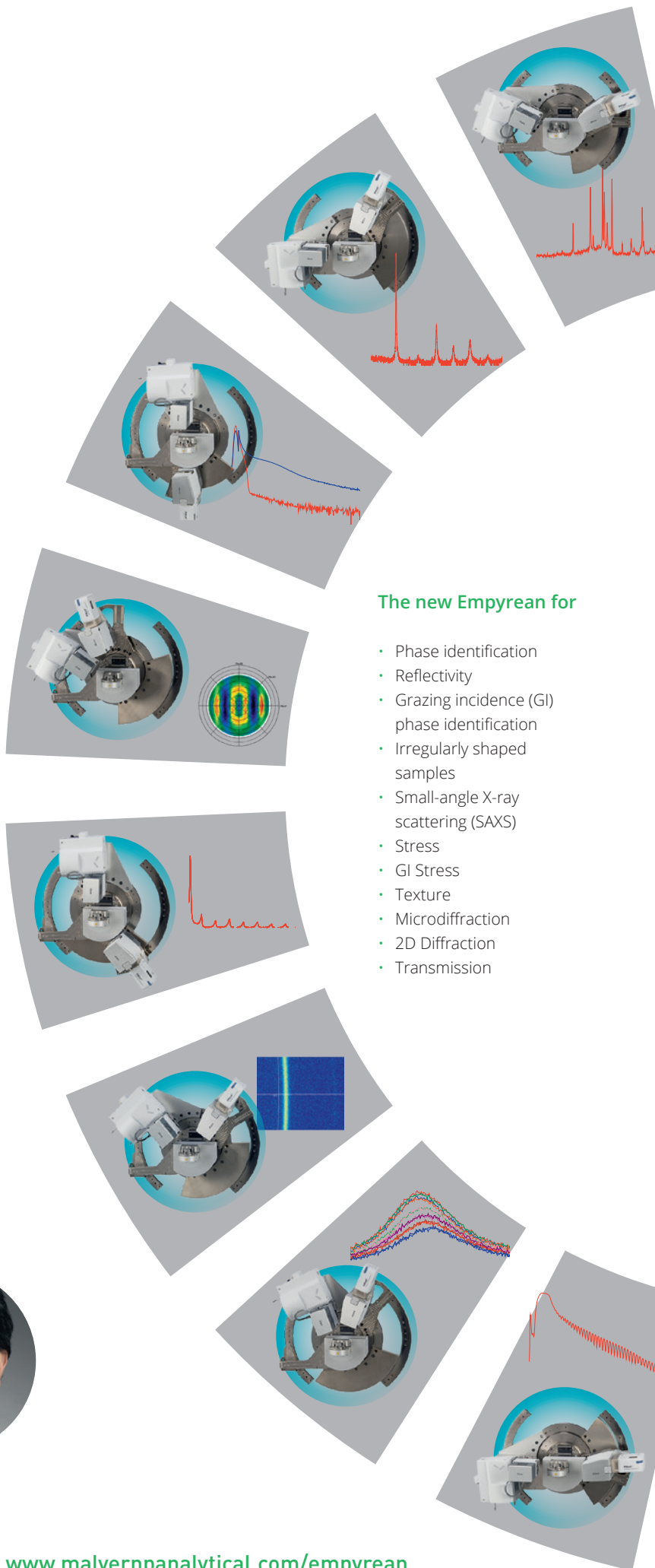
More experienced users will still enjoy our renowned proprietary concept of pre-aligned fast interchangeable X-ray modules (PreFIX), meaning that all optics can easily be exchanged without the need for re-alignment. As an additional benefit, all optics are now automatically recognized by the system, simplifying the exchange and eliminating errors due to incorrect reporting of measurement conditions.

The new Empyrean will solve many worries of non-expert XRD users by ensuring that they obtain the best possible data when using the new MultiCore Optics. "We have not forgotten the expert user, though. Whoever wants to perform more advanced measurements, will still be able to do so, by employing our dedicated optics which can be fine-tuned to deliver even better data," explains Fabio Masiello, Malvern Panalytical's product manager for the Empyrean. "The new Empyrean is the perfect instrument not only for the expert but also for the beginner. Users can expect to spend less time in training activities and have absolute confidence in their data."

"The simplicity and accessibility delivered by the MultiCore Optics on our new Empyrean could increase utilization of diffractometers in the lab by around 35% and they help even inexperienced users to achieve good X-ray diffraction data."



Fabio Masiello
Malvern Panalytical Product Manager
Empyrean



The new Empyrean for

- Phase identification
- Reflectivity
- Grazing incidence (GI) phase identification
- Irregularly shaped samples
- Small-angle X-ray scattering (SAXS)
- Stress
- GI Stress
- Texture
- Microdiffraction
- 2D Diffraction
- Transmission

MEET THE NEW REVOLUTIONARY ZETASIZER PRO AND ZETASIZER ULTRA

On May 23rd, Malvern Panalytical's new Zetasizer® Pro and Zetasizer Ultra were launched via global webinar and simultaneously at the IACIS 2018 colloids meeting in Rotterdam. These instruments have been specifically created to increase confidence in decision-making within key analytical workflows associated with product development and quality control.

The Zetasizer Pro and Zetasizer Ultra, already the proud recipients of a prestigious Red Dot 2018 design award, are our flagship dynamic and electrophoretic light scattering instruments for the measurement of particle size distributions and zeta potential. These parameters may sound obscure, but they impact on all sorts of everyday products, from your morning coffee to the shampoo you wash your hair with, the paint you use to protect your garden fences, and the toothpaste you use. Of course, they also play key roles in not-so-everyday applications, such as helping to determine the shelf-life of cancer treatments and optimizing the design of new nanomaterials.

Innovation through dialogue

Malvern Panalytical has a proud history of developing and manufacturing practical yet innovative technologies which help our customers across the globe solve the challenges associated with maximizing productivity, developing better quality products and getting them to market faster. Our Company's President, Mr. Paolo Carmassi, is clear in his view of how the company is evolving to deliver even more value: "Our vision is to create an environment where customers talk to us about their real problems, where they see us as partners, collaborators, and experts in the detail of their business."

Darrell Bancarz, Nanomaterials Product Manager, confirmed, "The Zetasizer Pro and Zetasizer Ultra are the result of extensive customer understanding, research and testing, which has enabled

us to develop a more intelligent and intuitive way of making light scattering measurements. We've introduced capabilities that halve sample preparation and measurement times, while providing more stable and reliable results. The application of revolutionary new measurement approaches delivers extended materials insight, enabling true product and process optimization."

Pavel Abdulkin, Head of Particle Works, Blacktrace Holdings Ltd., which develops and manufactures high-performance nanoparticles and microparticles, was clear about the impact of the new technology on his work: "The Zetasizer Ultra has dramatically increased our sample throughput, enabling us to accelerate our particle development cycle from 12 months to around a month."

Groundbreaking software

The Zetasizer Pro and Zetasizer Ultra systems are controlled by groundbreaking ZS Xplorer software, introducing new sample-centric workflows which make method design and data analysis more straightforward for new users, while preserving full accessibility for users experienced in the application of light scattering methods.

For the first time, new analysis capabilities answer key issues associated with the presence of dust or aggregates when applying dynamic light scattering for particle and molecular characterization, filtering these from the results to give a truer overall picture of the sample. This is supported by integrated, deep-learning



empowered data quality guidance, offering the user instant feedback on their results and providing actionable advice on how to improve poor quality data.

A key differentiator of the Zetasizer Ultra is its patented Multi-Angle Dynamic Light Scattering (MADLS®) technology, which automates multiple-angle size measurements, providing higher resolution and more complete particle size distributions. MADLS also enables calibration-free particle concentration analysis for a wide range of materials, resolving the individual concentrations of different size populations.

The new disposable capillary sizing cell provides the ultimate in non-destructive, low-volume (3 µL) analysis, extending the upper size range to 10 µm and delivering high-quality data while reducing cost. The Zetasizer Ultra's speed and ease of use are uncompromised by these unique and powerful capabilities, making it the most advanced light scattering system available.

Zetasizer systems are used across many industry sectors worldwide, delivering value in the control and optimization of processes, and the improvement of product quality, stability and performance. They are ubiquitous in academic settings, indispensable in a wealth of application spaces and referenced in tens of thousands of peer-reviewed publications. To find out more about this cutting-edge technology, please visit www.malvernpanalytical.com/zetasizer.

“The Zetasizer Ultra has dramatically increased our sample throughput, enabling us to accelerate our particle development cycle from 12 months to around a month.”

Pavel Abdulkin
Head of Particle Works,
Blacktrace Holdings Ltd



THE HISTORY OF X-RAY TECHNOLOGY

1945 - 1972

A SIX-PART SERIES

Although the principles and applications of X-ray fluorescence and X-ray diffraction had been well-known in the scientific world since the 1920s, these techniques were only used by experienced scientists and researchers. The breakthrough into analytical labs came with young scientist Herbert Friedman's introduction of the counter tubes for X-ray diffraction experiments.



1895 - 1917



1917 - 1945



1945- 1972



1972 - 2002



2002 - 2017



2017 - ...

NORELCO, A SUCCESS STORY*

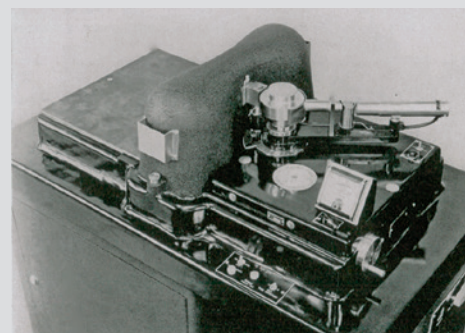
Herbert Friedman and the Norelco Ninety Degree Diffractometer

Until 1940, X-ray diffraction patterns were only recorded on film, mostly using the Debye Scherrer camera. Although X-ray detectors were available at that time, their reliability and accuracy were not sufficient for routine use. In 1940, scientist Herbert Friedman from the Naval Research Laboratory in Washington, D.C. (USA) worked on the accurate determination of the crystallographic orientation of quartz crystals used for radiocommunication. As these film-based experiments took rather long, Friedman replaced the film by a Geiger counter, which considerably sped up his measurements. Herbert Friedman obtained a patent for this setup in 1942.



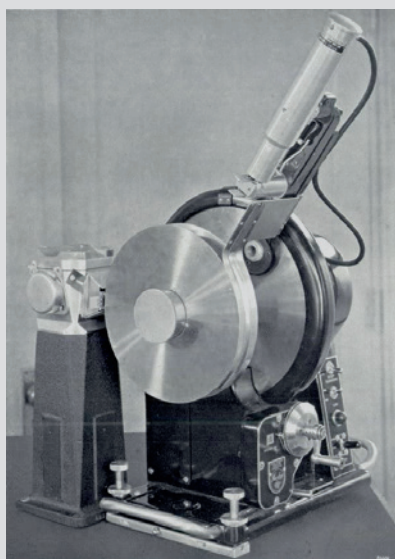
Herbert Friedman¹

To further develop his system for quartz crystal alignment, Friedman was looking for a collaboration with manufacturers of X-ray equipment and invited them to Washington. In his memoirs, Friedman remembered: "The only one that wanted to talk seriously with me about it was the Philips company". The collaboration resulted in the Norelco Ninety Degree Diffractometer, introduced in 1945 to the market by Philips as the world's first commercially available X-ray diffractometer.



The Norelco Ninety Degree Diffractometer

* Norelco stands for **N**orth **A**merican **P**hilips **E**lectrical **C**ompany, Philips' brandname in North America. The name Philips could not be used until 1981 due to legal reasons.

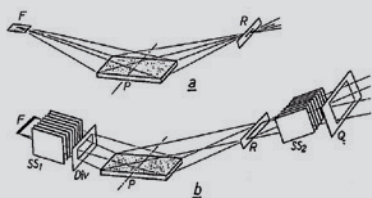


The PW1050 X-ray diffractometer

Friedman's diffractometer for the alignment of crystal orientation was a horizontal diffractometer with vertical sample position, which is less suitable for powder measurements.

In the Philips Research Laboratory at Irvington-on-Hudson (NY, USA), mineralogist Bill Parrish was working on the next generation of diffractometers. Philips had developed a water-cooled X-ray tube for vertical use offering Parrish the opportunity to develop a compact vertical diffractometer. Its radius was 173 mm, the standard diffractometer radius for a long time.

Another innovation by Bill Parrish was the use of the X-ray tube's line focus in combination with Soller slits to decrease the axial divergence. This way, Parrish improved the classical Bragg-Brentano geometry to the line focus application that we still use nowadays.



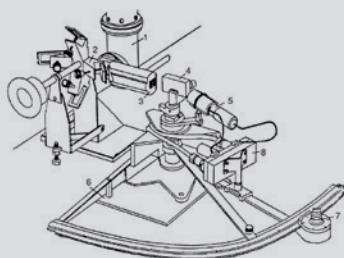
Classical Bragg-Brentano geometry (top) vs. Parrish's improved line focus geometry²

Parrish's diffractometer was put on the market in 1948. After some improvement cycles, Philips introduced it as PW1050 X-ray diffractometer in 1952 and sold over 10,000 copies of this system worldwide.

The X-ray fluorescence unit

During experiments with the Norelco Ninety Degrees Diffractometer, Herbert Friedman discovered that the background intensity of measurements with copper-radiation on iron-containing samples was unusually high. The use of a high-power X-ray tube and sensitive detector revealed the background radiation, which could hardly be seen in previous measurements on film. Friedman got the idea to use X-ray fluorescent radiation as means to determine the elemental composition of a sample.

Together with his colleague Verne Birks he transformed a diffractometer into a spectrometer by placing an analyzer crystal on the sample position and a new sample holder between tube and crystal – a simple spectrometer for which Friedman obtained a patent. In 1948 North-American Philips brought the spectrometer to the market as a fast and non-destructive analytical tool: the world's first commercially available spectrometer.



The X-ray fluorescence spectrometer by Friedman and Birks³

The Norelco Application Laboratory

When the first XRD and XRF systems were introduced to the market, the techniques still had to find their way into the industrial and scientific laboratories. To demonstrate their equipment, North-American Philips established a test laboratory in their premises in Mount Vernon (NY, USA). In a 1953 publication, they said about their lab: 'The engineers at Mount Vernon are constantly seeking to formulate experimental data and correlate the findings of the Philips'

Laboratories for product improvement and wider applications. Our users are continually discovering new methods which widen the scope of the X-ray diffraction equipment.'

Malvern Panalytical's application labs in the Netherlands, Japan, China and the Americas are direct successors of this Norelco Application Laboratory.

Norelco Reporter

After the introduction of the first commercially available X-ray diffraction and fluorescence units, Philips wanted to inform their steadily increasing customer base about the developments of analytical X-ray equipment. The Norelco Reporter was established as North-American Philips' customer magazine in September 1953. It contained product information from Philips next to application stories, written by users. Information about analytical X-ray equipment was later complemented by articles about electron microscopy.

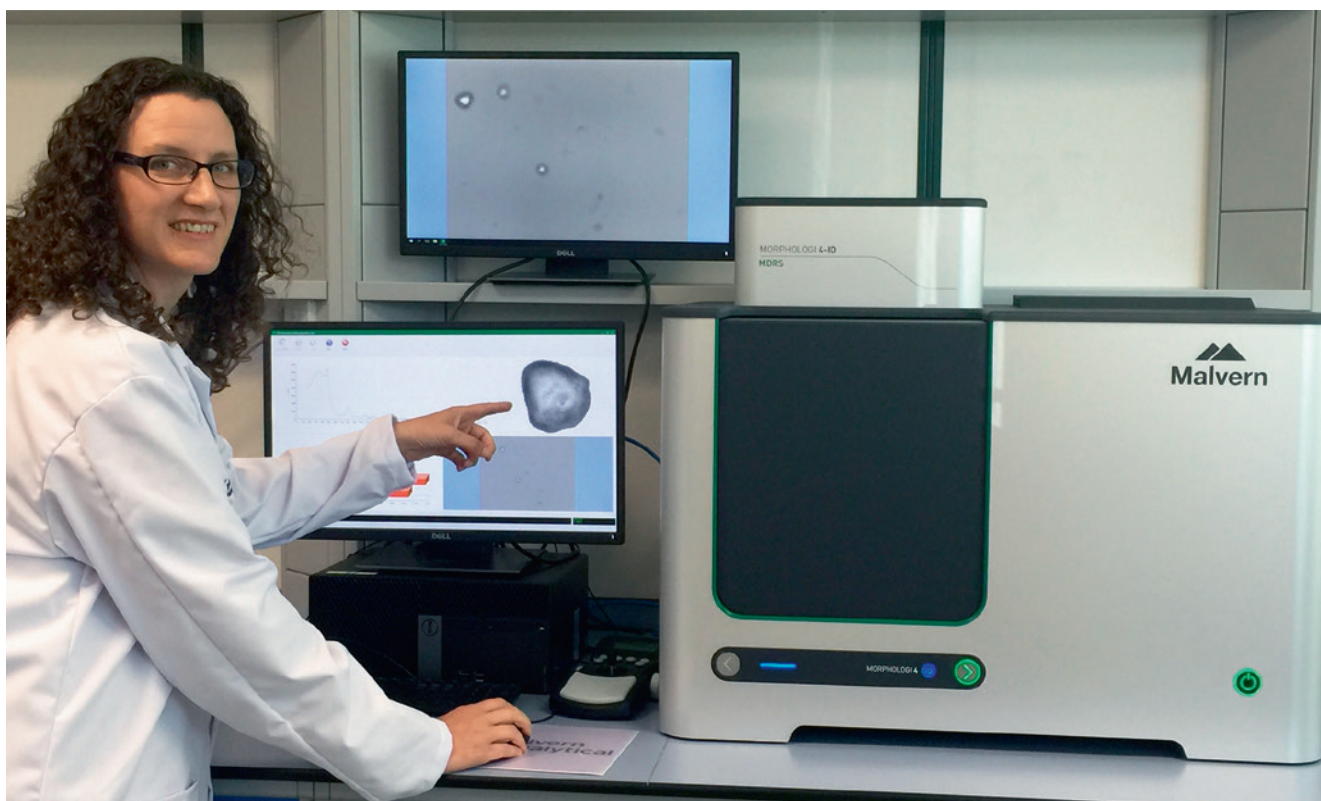
The Norelco Reporter was published between 1953 and 1985 and the articles were usually of such a high level that many scientists collected the issues over the years. Malvern Panalytical's XPress magazine, published since 1993, is the successor of Norelco Reporter.



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- 2) W. Parrish in 'Crystallography in North America'. Edited by D. McLachlan Jr. & J. P. Glusker. New York: Am. Cryst. Ass, 1983
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THE NEW MORPHOLOGI 4 RANGE: **IMAGE IS EVERYTHING!**



Debbie Huck-Jones is Malvern Panalytical's Product Manager for Analytical Imaging, and led the global launch of the new **Morphologi 4** and **Morphologi 4-ID** automated static imaging instruments in February. These systems deliver fast, high-definition particle characterization which is used across a huge range of industries, adding value to product development, troubleshooting and QC. They are particularly useful in analytical environments, where a deeper understanding of a process and/or sample is required.

Tell us a little more about yourself, and how you ended up in the role of Product Manager for Analytical Imaging at Malvern Panalytical.

Despite the thousands of miles I now travel every year, I was born and raised 9 miles away from Malvern Panalytical's UK headquarters. My Master's degree in Chemistry from the University of Exeter included European study, and I spent a year in Montpellier, France. My PhD on the synthesis and characterization of liquid crystals was jointly awarded by the University of Exeter and the Université Louis Pasteur in Strasbourg under *co-tutelle*, meaning I split my time between the two.

In 2005, I came to the company then known as Malvern Instruments as a Product Technical Specialist for our imaging products, later moving into the role of PTS Supervisor for imaging and laser diffraction. What was great about both roles was that I could work closely with our customers and existing users all over the world, providing applications development and support for the Morphologi and other imaging instruments. I also got to work with our imaging development team, supplying them with applications feedback to continuously improve our products in line with our users' requirements. This experience made my 2014 step into the role of Product Manager for our analytical imaging technologies a natural

one – now I sit between multiple teams, leading the development of our products with a constant eye on what our customers need and how analytical requirements are evolving.

So, looking back on the launch of the Morphologi 4 and Morphologi 4-ID, what can you tell us about the process which led to their global release?

Although the development of these products goes back a little further, the real process of preparing for launch took around 3 years. It was started before Malvern Panalytical's January 2017 merger, and although we've always been a customer-

centric company, our mission became very clearly defined at that point. We started to look even more closely at maximizing the value we deliver, clarifying the root causes of analytical requirements to understand how we could develop a unique offering with positive economic impact.

In the case of the new Morphologi 4 range, we had an additional project driver in that we knew some parts used in the Morphologi G3 and Morphologi G3-ID would soon become obsolete. We decided that it was an ideal time to evolve the systems to include groundbreaking new hardware and software features that really spoke directly to what our users were requesting. So, we took the unprecedented step of integrating the Raman spectrometer into the same enclosure as the automated microscope in the Morphologi 4-ID. We made the software easier and quicker to learn and use, which was helped by the development of the Sharp Edge tool. Additionally, this assists with characterization of lower contrast samples such as protein aggregates.

The whole time, we worked alongside our customers to make sure we were delivering exactly what they wanted. This process takes a long time – it's not as simple as receiving a request and immediately implementing it. We needed to ensure that requirements were representative of our larger customer base, and make changes in a measured, practical way so that we knew they worked properly and seamlessly. We also kept a careful watch to make sure that customers who own a previous generation of the Morphologi system were assured of continuity with their data and methods.

What really excites you about the new Morphologi 4 range?

I love the whole new look and the enclosure of the instruments! We were awarded both a Red Dot 2018 award and an Industrie Forum 2018 award for the design of the Morphologi 4 range, and this is mainly due to the creation of a sleeker, more modern look. The work area is now enclosed behind an automatic sliding door. In addition to avoiding light and dust contamination, this, along with the necessary interlocks, makes the Morphologi 4-ID system Class 1 for laser safety. When the door is open, the measurement area is automatically illuminated, which facilitates work on the microscope stage.

The Sharp Edge function, improved image quality and extended range of the new instruments really push the boundaries of analysis, and they extend the available application spaces for Morphologi. Morphologi systems are now found throughout academia and industry, in sectors as diverse as forensic analysis, battery material development and manufacture, pharmaceutical bioequivalence studies, additive manufacturing, building material development and mining! The list really does keep growing week by week, and we find that scientists in all disciplines love the simplicity and clarity offered by Morphologi systems.

And I have to mention the improvements to measurement and data analysis times. These new instruments are very fast! When you add that to the ease and flexibility of the workflows, the value these new systems deliver is really amplified.

Now that the systems have been launched, what's next for the Morphologi range and their Product Manager?

A product launch is only the beginning of the story! New products need support from their product managers at different times and in various ways, all over the world. What I'm really enjoying is the time spent, not only with our customers, but also with our specialists across the globe. The feedback I've had from them has been great, particularly with respect to the 'out of the box functionality' of the new systems – this really helps them at exhibitions and seminars, and also on installation.

And the future for imaging and Morphologi generally? There is an exponentially expanding role for imaging in many different applications, including those mentioned above, but we are also excited about the possibilities in newer areas of research such as the analysis of microplastics in drinking water, and many others. Please do get in touch if you think you might have a new application you want to test using the Morphologi 4 or Morphologi 4-ID!



"The whole time, we worked alongside our customers to make sure we were delivering exactly what they wanted."

Debbie Huck-Jones
Malvern Panalytical Product
Manager, Analytical Imaging

COMBINING FORCES FOR THE ENVIRONMENT

With ever-increasing industrialization, the protection of our environment is essential; we need to do everything we can to protect our Earth. One starting point is a fast, precise and reproducible analysis of those resources we return to nature after use, such as wastewater and emissions to the air.

Wastewater management

Over the last two years, companies in Asia were fined for disposing of used industrial waste tainted with chemicals and heavy metals into the water. For example in Vietnam, fish carcasses washed up on the beaches of Hà Tĩnh and many fishermen lost their livelihoods. This clearly also had a lasting effect on water safety. Although these companies have received fines, their waste management issues still affect the public and ecosystem.

Careful monitoring of wastewater before it is disposed of can save a lot of money (fines range from millions to billions of dollars) and can prevent damage to the environment, avoiding the need to spend on recovery efforts.

Malvern Panalytical supports companies with analytical solutions which ensure environmentally responsible disposal of used industrial water.

- Prevent heavy metals from getting into your process: careful in-bound QC elemental analysis for heavy metals using our latest **Epsilon 4** benchtop EDXRF spectrometer.
- Monitor factories' effluent streams: online monitoring of your wastewater for heavy metals before disposing into the environment using **Epsilon Xflow**. Immediate counteractions can be taken to prevent environmental damage.
- Treating water with contaminants: Add the right dose of coagulants by carefully monitoring the zeta potential of particles during your flocculation and sedimentation process. Take a look at our **Materials Talks** blog to see how our **Zetasizer WT** was used after a fire to treat the ash in Canada's waters.

Monitoring the air we breathe

Outdoor air pollution can have many sources, both anthropogenic (power generation, vehicle emissions, industrial and agricultural emissions, etc.) and natural, for instance volcanic eruption, wind erosion or wild fires. A key area of concern is the Suspended Particulate Matter (SPM), which can have adverse effects on health, particularly in the particle size range under ten microns. Depending on their size, they have the ability to penetrate deep into lungs or permeate throughout the body and even into the brain.

Particulate matter has been classified by the World Health Organization (WHO) as a Group 1 carcinogen alongside tobacco smoke and asbestos¹, and particles <2.5 micron have been attributed to over 3.2 million premature deaths annually².

Many countries and regions of the world have enacted legislation setting air quality standards. The US Environmental Protection Agency (US EPA) published a comprehensive method (IO-3.3) for determining the elemental concentrations of 44 elements on air filters using energy dispersive X-ray fluorescence (EDXRF)³.

When this method was developed in 1999, measurements by floor-standing instruments took over 4 hours per sample to achieve the detection limits specified in the EPA method. Nowadays, the recently launched Malvern Panalytical Epsilon 4 EDXRF benchtop instrument has the capability to achieve fully compliant results in only 45 minutes per sample. Air filters can be loaded into the spectrometer with only minimal sample preparation and remain intact for further analysis such as weighing and analysis of hydrocarbons. For all these reasons, EDXRF is less labor-intensive compared to alternatives such as ICP and AA.

Learn more about elemental analysis of air filters according to the EPA method IO-3.3 using the Epsilon 4 in an application note on our [website](#).

"The importance of monitoring and testing of air quality is demonstrated by the involvement of both ASTM International Committee D22 (established in 1951 and representing 24 countries) and ISO Technical Committee 146, both focusing on air quality. Global harmonized monitoring and testing is the start of understanding where we stand and deciding internationally which improvement measure to implement."

Taco van der Maten

Malvern Panalytical Segment Manager Oils, Chemical, Polymers / Vice Chairman of the ASTM Board of Directors



Concern for respirable silica

Airborne silica particles pose a carcinogenic threat especially for foundry workers, at stonecutting, quarry work and tunnelling, even at concentrations as low as $50 \mu\text{g}/\text{m}^3$. The maximum permissible exposure limit of respirable crystalline silica (RCS) at a workplace is under constant scrutiny and there is a continuous push to improve limits of detection and quantification while keeping a reasonable measurement time. X-ray diffraction (XRD) is the method of choice for these demands and can even distinguish between the common polymorphs of silica (quartz, cristobalite and tridymite).⁴

Recent advances in optics design have led to great improvements of the low limits of detection for XRD, and in the case of RCS analysis, greatly reduced measurement time to as fast as 90 minutes to measure four reflections of quartz. Malvern Panalytical's XRD instruments combine high sensitivity and speed of detection and offer a robust turnkey solution for the quantification of respirable silica. Details can be found on our [website](#).

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REVOLUTIONIZING THE ANALYSIS OF BASE METAL SULFIDES



Sulfide ores are important sources for several elements, such as Cu, Zn, Pb, Ni, Co, Mo, Bi, Sb, Cd, Ag, Au, Se and Te. Industrial production of base metals and associated minerals makes use of, for example, CuFeS_2 (chalcopyrite), ZnS (sphalerite), FeAsS (arsenopyrite) and other sulfides. In order to obtain maximum yields from a deposit, it is crucial to know the concentrations of the valuable elements in ores and concentrates at various stages of the mining process, from prospecting to exploration and processing.

Traditional analytical methods are based on wet-chemical titrimetric and gravimetric techniques, which are considered reference techniques as described in various industry norms. However, for routine analysis, other methods are generally preferred. Compared to ICP-OES and AAS, X-ray fluorescence (XRF) is an advantageous technique because it does not require any digestion using hazardous chemicals, which prevents chemical waste and saves time. Due to the high variability in the mineralogy of the materials, sample preparation by fusion is required to achieve optimal accuracy. Additionally, reliable calibration materials are needed.

Previously, these requirements for the precise XRF analysis of metal sulfide ores and concentrates posed a challenge because of the range of concentrations to be determined. While base metal ores contain 0.5–3% valuable minerals, the content of valuable elements is increased to 20–50% after concentration. At the same time, the total amount of sulfides in these concentrates

can be as high as 80–90%. It is difficult to find certified reference materials covering such elemental and concentration ranges, if they are available at all, which made calibration a difficult and costly step. In addition, the sample fusion step is critical as all elements, including the sulfur, need to be retained in the bead. Due to the high metal and sulfide content, complete sample oxidation and dissolution in the bead, whilst also preventing damage to the expensive platinum ware, is not trivial.

The universal need of the mining industries for quick, reliable and accurate sulfide analysis was the trigger for Malvern Panalytical to develop a novel and complete analytical solution based on XRF and fusion. This method revolutionizes the analysis of these challenging materials. It not only alleviates the difficulties traditionally encountered in XRF analysis of these materials, but also greatly simplifies the entire analytical process, adding significant value over alternative methods.

Uwe König, Malvern Panalytical Global Mining Manager, explains: "Mining companies will be able to increase their yields when employing this fast and accurate analysis of mined material."

The method uses synthetic calibration standards, covering 21 elements in concentration ranges typically encountered for base metal sulfide ores and concentrates. It allows the easy addition of elements and/or an extension of concentration ranges if needed. The solution comes with a ready-to-use flux and fusion method, ensuring complete oxidation and retention of all elements in the bead. The preparation method is designed for Claisse electric fusion systems like **TheOx** and **Eagon 2** while the analytical program can be deployed on **Zetium** or **Axios** spectrometers. If desired, the method is fully automatable.

Employing XRF for the process critical-analysis of metal sulfides offers several advantages over alternative analytical approaches:

Speed: A sample's analysis covering all elements takes less than 45 minutes from weighing of the sample to receiving the result. The same or more time is normally required to determine the concentration of one element only when using classical methods.

Complete sample analysis: More than 20 elements are reported at the same time with high accuracy and precision. This is especially relevant for the optimization of processes like material grinding and flotation.

Accuracy: Accuracy of the analytical results is the same or better than that required by the norms based on classical methods.

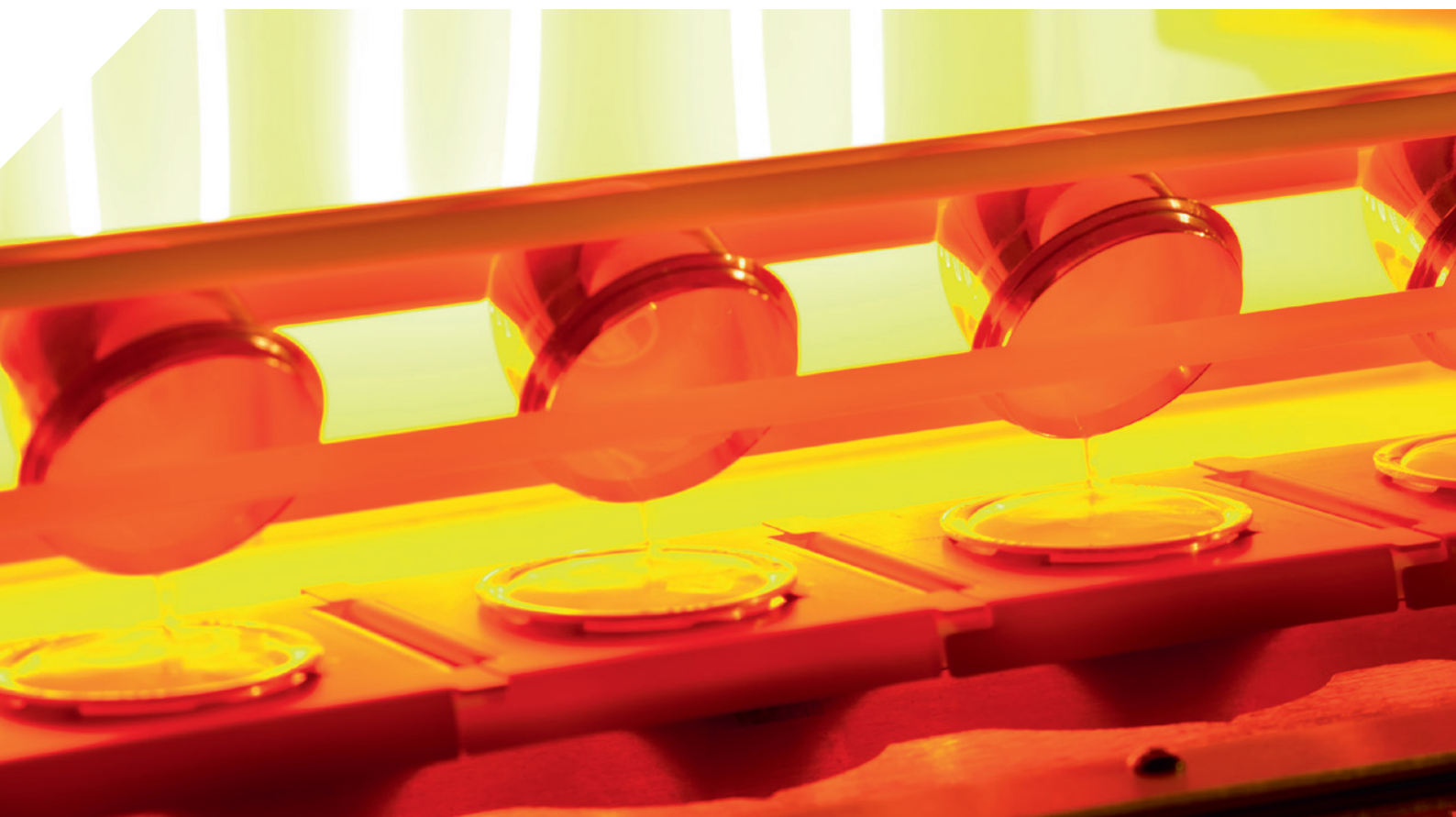
Simplicity: The complete method does not require highly qualified personnel, in contrast to other instrumental and wet-chemical techniques.

Contact us at expertise@malvernpanalytical.com if you want to know more about this method or other Malvern Panalytical analytical solutions.



"Our new complete solution for the precise analysis of base metal sulfides finally conquers the challenge this task posed in the past."

Marco van der Haar
Malvern Panalytical Business
Development Manager Expertise

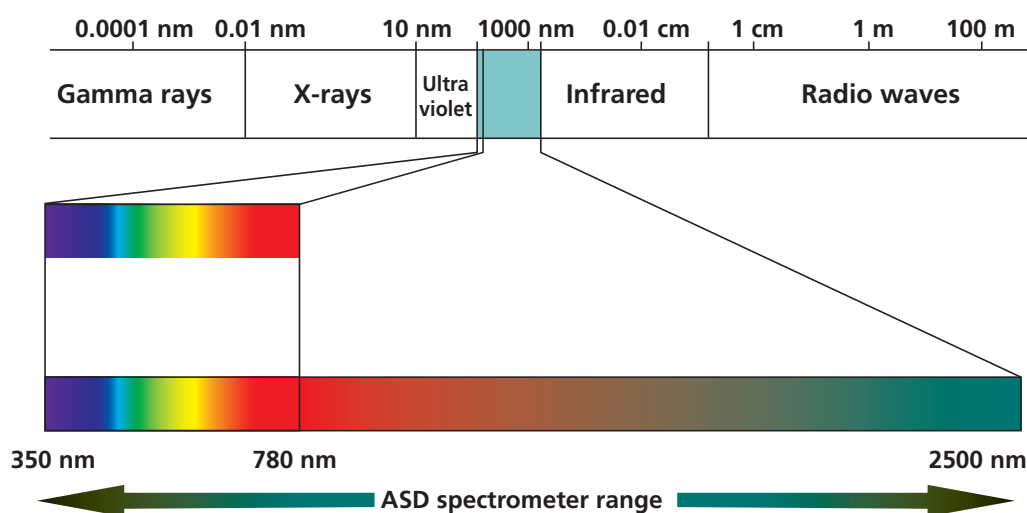


OPTICAL SPECTROSCOPY FOR INDUSTRIAL AND REMOTE SENSING

The use of VNIR (visible near-infrared) optical spectroscopy allows real-time measurement of materials in the field, in the lab, near-line, and on-line. ASD, a brand of Malvern Panalytical, offers a range of such optical spectroscopy instruments that are portable, rugged, research-grade, non-invasive and non-destructive, require little to no sample preparation, and enable rapid and cost-effective real-time measurement of data.

VNIR technology

Spectroscopy involves the study of how light energy interacts with materials - it is either reflected, transmitted, or absorbed. How the material interacts with light energy can be diagnostic of the material. Spectroscopy uses this principle and combines it with the ability to sample very small wavelength intervals and combine them into a continuous spectral signature.



ASD spectrometers utilize the optical spectroscopic region of the electromagnetic spectrum (from about 350 to 2500 nanometers). Reflectance spectra can then be used to quickly determine a material's properties without altering or even coming in contact with the sample. Capable of examining irregular surfaces with the same ease as a carefully prepared sample, these instruments are non-destructive, and require little or no sample preparation. They can also be used to analyze multiple constituents in a single scan.

This highly flexible analysis can be used for a broad range of research

and industrial process applications. Long considered a staple technology in earth remote sensing, reflectance spectroscopy has become popular within industrial markets as a cost-effective tool for measuring materials to obtain actionable information to help optimize processes, manage costs and improve research.

Most ASD instruments measure over the 350 to 2500 nm wavelength range, the exception being the [FieldSpec® HandHeld 2](#) which measures from 325-1075 nm. Unlike other measurement tools that use X-ray or ultraviolet, light in this optical wavelength range is non-hazardous.

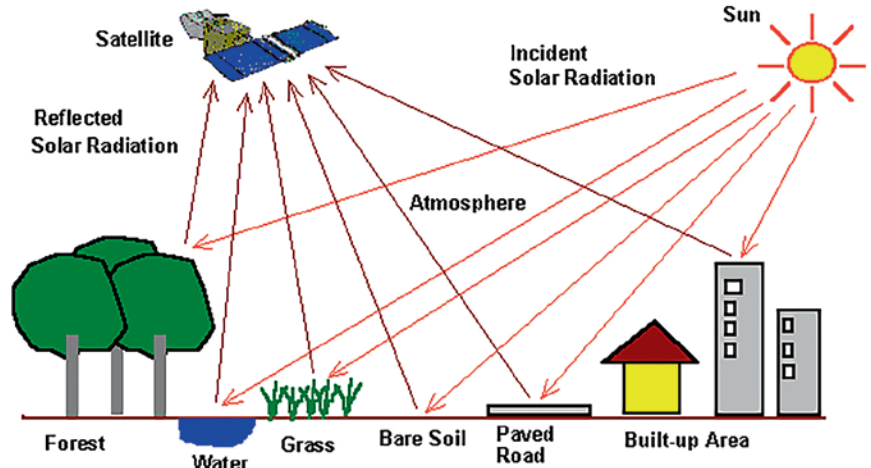
This wavelength range includes part of the ultraviolet range (350-400 nm) and the entire visible range (400-700 nm). The term(s) used for the remainder of this wavelength range (i.e. 700-2500 nm) varies by application area: remote sensing and mineral exploration users use the terms near-infrared for the 700-1000 nm range and short-wave infrared for the 1000-2500 nm range, while users in industrial applications such as food and pharma use the term near-infrared to indicate the entire 700-2500 nm range.

Remote sensing

Remote sensing is defined as 'the science and art of identifying, observing and measuring an object without coming into direct contact with it.'¹ This is a broad term that can include everything from reading a book to medical imaging to using an ASD optical spectroradiometer – in all cases you are getting data and information without being in physical contact with the sample.

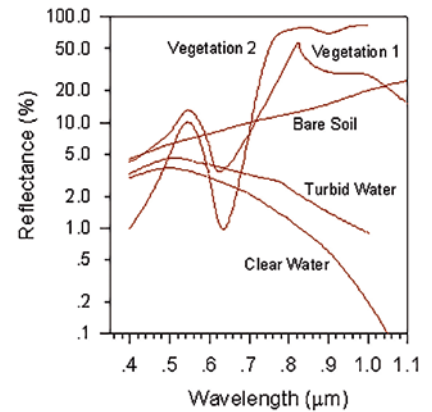
Remote sensing can aid in mineral exploration, archeological studies, agricultural analysis for water and fertilizer optimization and many more.

ASD instruments, specifically the FieldSpec range, are often used for passive optical remote sensing, which depends on the sun as a source of illumination. Visible, near-infrared and short-wave infrared sensors form images of the earth's surface by detecting the solar radiation reflected from ground targets. These ground targets can be differentiated by their spectral reflectance signatures.



Optical remote sensing²

A spectral signature is like a fingerprint. Different surface types such as water, bare ground and vegetation reflect radiation differently in various channels. Essentially, the configuration of the reflectance spectra allows an operator (using comparison tools, like a spectral library) to recognize a type of material.



Reflectance spectrum of five materials: clear water, turbid water, bare soil and two types of vegetation.²

References

1. Evelyn L. Pruitt, US Office of Naval Research
2. Images sourced from <https://crisp.nus.edu.sg/~research/tutorial/optical.htm>



ASD (formerly Analytical Spectral Devices) is part of Malvern Panalytical and delivers optical spectroscopy instruments for the remote sensing, mining and other industrial markets. They complement and extend the company's offering towards scientific and industrial customers adding a new contiguous product line with portable, handheld, benchtop and on-line products.

ASD optical spectrometer and spectroradiometer instruments have many diverse application uses and can be employed in various market sectors, such as environmental (**FieldSpec® line**), mining (**TerraSpec® line**) and food & pharma (**LabSpec® line**) and are occasionally used in forensics, cosmetics, and building materials.





PROTECTING CUSTOMER DATA

The data our solutions provide is precious. Whether it is particle size or crystal structure, binding affinity or elemental analysis, our solutions provide data outputs which generate insights needed to overcome business challenges. Data leadership is Malvern Panalytical's business; in helping us to deliver this, we have received data from hundreds or thousands of the greatest data processors in the world - our valued customers!

Malvern Panalytical's organizational headquarters are located within the EU, and so the organization abides by the regulations of EU law on data protection. The small amount of personal data that our customers have provided to us in the past has helped us to foster lasting relationships, build communities and spread knowledge that sparks innovation. The updated European Union ('EU') law on data protection, 'GDPR' (General Data Protection Regulation), addresses the export of personal data outside of the EU and requires all of us to be informed about who holds our data, how they use it and why.

Having helped thousands of customers create the systems and processes to generate materials analysis data in compliance with industry regulations, Malvern Panalytical is proud to have applied GDPR regulatory compliance to our own business. Those of you who have subscribed to our email communications in the past are now being asked to re-confirm your subscription, thus consenting to receive emails from us - so that we can continue the conversation and keep in touch regarding upcoming Malvern Panalytical user events, webinars, etc..

This is a small but essential task which ensures we only use our customer's personal data with their explicit permission. We hope that all of our customers will opt-in and consent to our new communication efforts so that together, we can set new trends, build on best practices, and collaborate to improve processes and keep one another informed.

Our new email experience is easy to access, and we will be using the data you provide to ensure that the experience you receive is most relevant to your region, industry and interests. Our mission is not to bombard or distract, but rather to inform and encourage you to be successful with our solutions and make the most from opportunities to connect with the community.

To sign up, and/or consent to receive our email communications, please visit

www.malvernpanalytical.com/register.

Thank you, and welcome to Malvern Panalytical!

PINPOINTING MAJOR CAUSES OF ERRORS IN XRF ANALYSIS

Are you still struggling to produce reliable and repeatable results with X-ray fluorescence (XRF) analysis?

Analyzing samples prepared as glass disks with an XRF spectrometer remains the fastest and most convenient method for the chemical analysis of solid samples. However, the final results obtained with this technique can be impacted by many interrelated factors such as reference materials, the calibration of the instrument, preparation of the glass disks and compatibility between the fusion instrument and the XRF spectrometer.



Our whitepaper describes the root causes of analytical errors and explains how they can be addressed directly at the source with the help of just one supplier involved in both designing and executing the entire analytical process. The paper also includes a real case we recently encountered and were able to solve to the customer's satisfaction.

The publication can be found on our website: www.malvernpanalytical.com/fusionWP

PHARMACEUTICAL CHALLENGES AIDED BY A LABORATORY POWDER X-RAY DIFFRACTION SYSTEM

The pharmaceutical industry is on a constant quest to develop high-quality stable and safe products while accelerating their speed to market. Powder X-ray diffraction is an ideal technology to tackle this challenge because it allows the determination of many relevant parameters in a non-destructive way and delivers very precise and reproducible results.

In a recently published whitepaper Malvern Panalytical specialists describe several applications relevant for the pharmaceutical industry such as improved limits of detection of polymorph traces due to advances in optics, the determination of amorphous content with greater accuracy, *in situ* crystallization studies and the determination of

protein shape and stability. All these and more applications can be performed on a single X-ray diffraction platform.

The whitepaper shows examples of the mentioned applications and gives measurement details. It can be found on our website www.malvernpanalytical.com/PharmaWP.



COLOPHON

Please send your contributions, suggestions and comments to info@malvernpanalytical.com

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Want to visit us at an event? malvernpanalytical.com/events shows a list of events where you will find us. We are happy to welcome you!



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