PRESS

ISSUE **3/2018**

COLLABORATING FOR FAST AND DETAILED POLYMER ANALYSIS

Malvern Panalytical
a spectris company

CELEBRATING A YEAR OF INNOVATION

Tanneke Reinders
Vice President Marketing,
Malvern Panalytical

Dear reader,

With 2018 fast coming to a close, it's a good time to look back on a fantastic year, whilst also keeping a firm eye on the future. Since my arrival here at Malvern Panalytical, I've had the pleasure of learning more about both the company and its specialists. What is particularly exciting is the strength and reach of our new company with its combined expertise, and the exponential increase in the possibilities we are discovering, to help customers create ever-better products and processes, more efficiently than ever before.

In this final issue of XPress for 2018, you'll read about the recent opening of our Application Excellence (Application X) Laboratory in Shanghai, a real milestone for Malvern Panalytical in China. This lab is designed to bring together MP applications specialists and our customers to find innovative new ways of collaboration and problem-solving. There are some great stories coming out of China showing the application of our solutions for environmental analysis, pharmaceutical development and manufacture, and battery production, amongst countless others.

The battery market is a very hot topic at present, with demands from the automotive and renewable energy sectors to create batteries which are safer, cheaper and longer-lasting, growing particularly rapidly. Our article on p.16/17 explains how X-ray analysis, light scattering, rheometry and image analysis work side by side to complement each other perfectly and provide unique capabilities throughout the battery development and manufacturing processes.

We've also enjoyed a fruitful partnership with Waters Corporation which has recently resulted in the combination of their ACQUITY Advanced Polymer Characterization (APC) system with our OMNISEC REVEAL multidetector module. This provides unrivalled speed and detail for polymer analysis, and opens new avenues for exploration which will ultimately result in better product performance. Read all about it in this issue!

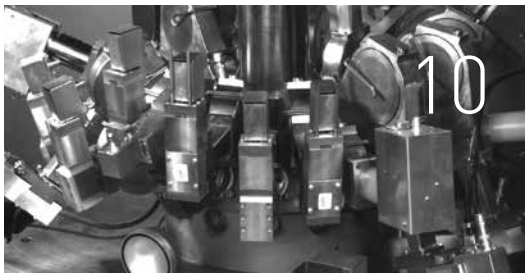
So, at the conclusion to a fast-paced and successful year, all of us here at Malvern Panalytical look forward to continuing exploiting our experience and innovative spirit to help our customers achieve excellence in diverse applications and unique challenges. Please continue to send in your comments, ideas and requests via info@malvernpanalytical.com.

We all wish you a successful close to 2018 and a prosperous start to the New Year!

With kind regards,

With kind regards,
Tanneke Reinders

IN THIS ISSUE



INVESTIGATING A 4.8 MILLION YEAR OLD GLACIER

The Urumqi (or Tianshan) Glacier No. 1 is located in the Tianshan Mountains 120 km southwest of Urumqi, capital of the Xinjiang Uyghur Autonomous Region in the far northwest of the People's Republic of China. It is the largest glacier near a major city in China and origin of the Urumqi River, one of the sources of water supply for the Urumqi area. Since the establishment of the Tianshan Glaciological Station (TGS) in 1959 the glacier has been closely monitored.

The Tianshan No. 1 Glacier has existed for 4.8 million years, but according to the latest reports it has only 50 more years to live. This can be compared to an 80-year-old man who is expected to die within 8 hours.The 'old man' witnessed volcanic eruptions, flooding, and nuclear bombing. It is melting into the Urumqi River towards a city with 8 districts, 51 ethnic groups and a population of 3.55 million, nourishing 14,000 km^2 of land in Urumqi. In recent decades, as the global climate has warmed, the glacier has melted more quickly.

Located at the source of the Urumqi River, the Tianshan Glaciological Station (TGS) of the Chinese Academy of Sciences is the only field station in China that specializes in glaciers as its main observation, test and research objects. The station has been a member of the World Glacier Monitoring Service (WGMS) since 1981 and is a representative observation site for inland glaciers in Central Asia. Over the last 40 years, a comprehensive and internationally-renowned database containing results of observations, experiments and research has been established with the support and assistance of the Chinese Academy of Sciences' research institutes.

Each year at the beginning and the end of the thawing period (the end of April to the end of August), the TGS researchers conduct a comprehensive study of the No. 1 Glacier, measuring parameters such as material balance, glacial thickness, glacier area, surface velocity, meteorology, hydrology, atmospheric aerosols, snow ice chemistry, glacier surface albedo and more. To achieve these measurements, TGS researchers use Malvern Panalytical's ASD FieldSpec® HandHeld 2 VNIR spectroradiometer, 2 ASD FieldSpec 4 Full Range spectroradiometers, and an ASD FieldSpec Dual spectroradiometer system to measure the snow's hyperspectral reflectance, irradiance and radiance of the Urumqi No.1 Glacier.

The ASD FieldSpec Dual spectroradiometer system inter-calibrates and synchronizes two full-range FieldSpec instruments to operate as a complete spectral data collection ecosystem. A base unit FieldSpec is dedicated to white reference measurements from a fixed reference panel, while the second FieldSpec unit provides mobile collection functionality for sample target spectra.

The two instruments communicate, inter-calibrate, and synchronize measurements via the mobile unit's laptop, Bluetooth and Wi-Fi. The systems enable collection of field reflectance spectra under less than ideal atmospheric conditions and separate the collection of white reference and sample radiance scans.

The collected data are used to study the dynamic process and spatial and temporal differences of the glacier's accelerated changes, to reveal the influencing factors and mechanisms and improve the parameterization scheme, aiming for a more realistic description of the physical mechanisms of key processes and reducing the uncertainty of glacial change simulation. The data provide a solid scientific basis for assessing the effects of glacial changes and response measures.

A video in Chinese language from the Xinhua News Agency shows the researchers at work on the glacier.

The team at work on the glacier, using the ASD FieldSpec

THE TIANSHAN GLACIOLOGICAL STATION (TGS)[1]

Built in 1959 by the former Lanzhou Institute of Glaciology and Geocryology of the Chinese Academy of Sciences (now the Cold and Arid Regions Environmental and Engineering Research Institute of the Chinese Academy of Sciences), TGS is located at the upper part and headwaters area of the Urumqi River.

The station is made up of a base and upper station with the base station located at the Urumqi Riverhead in a mountain valley at an altitude of 2130 m, covering an area of about 6000 m^2. Facilities comprise a meteorological observation field, a cold area plant testing land, laboratory, library, exhibition rooms, offices, and other research facilities. The upper station is located at 3545 m and covers an area of about 1000 m^2. It is 35 km away from the base station and serves as accommodation for the research teams and as storage for logistic equipment.

References

1) english.nieer.cas.cn/rs/sr/200908/t20090814_31900.html

ARGONNE NATIONAL LABORATORY ECOSPEC PROJECT LINKS HYPERSPECTRAL **REMOTE SENSING** AND **PLANT ACTIVITY**

As global temperatures continue to climb, research about climate change and its impact on the planet has taken on increased importance. Climate models simulate the interactions of important climate drivers, including atmosphere, oceans, land surface and ice, helping scientists to work through complicated problems and understand complex systems. One Illinois-based organization working to enhance current climate models is Argonne National Laboratory, a multidisciplinary science and engineering research center for the U.S. Department of Energy; their EcoSpec project investigates how plants respond and contribute to their surrounding local scale land surface processes.

Argonne's EcoSpec project team is led by Dr. Yuki Hamada and consists of remote sensing scientists, ecologists, electrical and mechanical engineers, and computer scientists[1].
Dr. Hamada's team strives to answer the big question of how interactions between the atmosphere and the land surface at a local scale relate to the interactions at regional and global scales and vice versa. Collecting a wide range of data types including hyperspectral remote sensing, meteorological measurements, and biological data should enable them to complement and enhance existing regional and global-scale climate models[2].

At present, field spectral data contribute to several earth observation applications including spatial information sciences. Here, the use of remote sensing for the analysis of environmental phenomena is scaled up - increasing from the local to the landscape scale. Identifying patterns and associations will help Argonne to link hyperspectral reflectance signatures with plant activity and improve the understanding of the relationship between ecosystems and climate.
The EcoSpec team has developed an optical tower system (a.k.a. EcoSpec system) to collect high temporal frequency hyperspectral reflectance measurements of land surfaces. The tower is equipped with multiple sensors that measure hyperspectral reflectance in the 350-2500 nm range, sky and land-surface temperatures, total incoming radiation, and light components (direct vs. diffused). Each sensor provides unique optical information about the land surface, including vegetation and exposed soil[3].

A spectrometer and RGB camera are housed in the box mounted on the top of the tower. Rotating around the tower 300 degrees, it collects spectral measurements and photos of the land surface at 12 positions. The system is autonomous and active from dawn to dusk throughout the growing season, streaming data wirelessly to a server at Argonne nearly every minute.

To track interactions between plants and their surroundings, and to better understand climate change through the spectral response of plants, theEcoSpec team uses an ASD FieldSpec® 4 Standard-Res spectroradiometer. In addition to the automatic optical measurements, weekly or semi-weekly canopy height, leaf moisture, and chlorophyll content are collected at several locations around the EcoSpec system. The team also uses the FieldSpec on the ground and in the field collecting the spectral data of leaves and leaf tissue to analyze leaf chemistry and pigments.
Argonne's EcoSpec team uses the collected data to develop a model for determining photosynthesis by integrating the respiration and hyperspectral data of the land surface. The patterns and associations will help to improve our understanding of the relationship between terrestrial ecosystems and climate.

Argonne is a multidisciplinary science and engineering research center, where talented scientists and engineers work together to answer the biggest questions facing humanity, from how to obtain affordable clean energy to protecting ourselves and our environment. Born out of Chicago's work on the Manhattan Project in the 1940s, Argonne leverages its Chicago-area location to lead discovery and to power innovation in a wide range of core scientific capabilities, from high-energy physics and materials science to biology and advanced computer science.

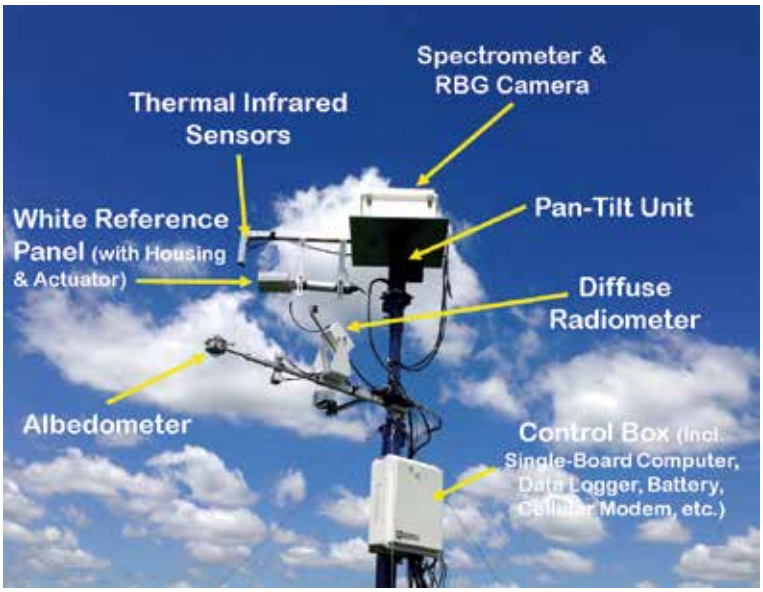
Argonne's EcoSpec project investigates patterns and associations between meteorological and biological measurements of the ecosystem corresponding to photosynthesis and

Argonne NATIONAL LABORATORY

effects of temporally varying factors (such as shadow) limiting photosynthesis and respiration of plants. **Their findings provide an opportunity to investigate how intra-annual variations in those factors affect ecosystem fluxes.**

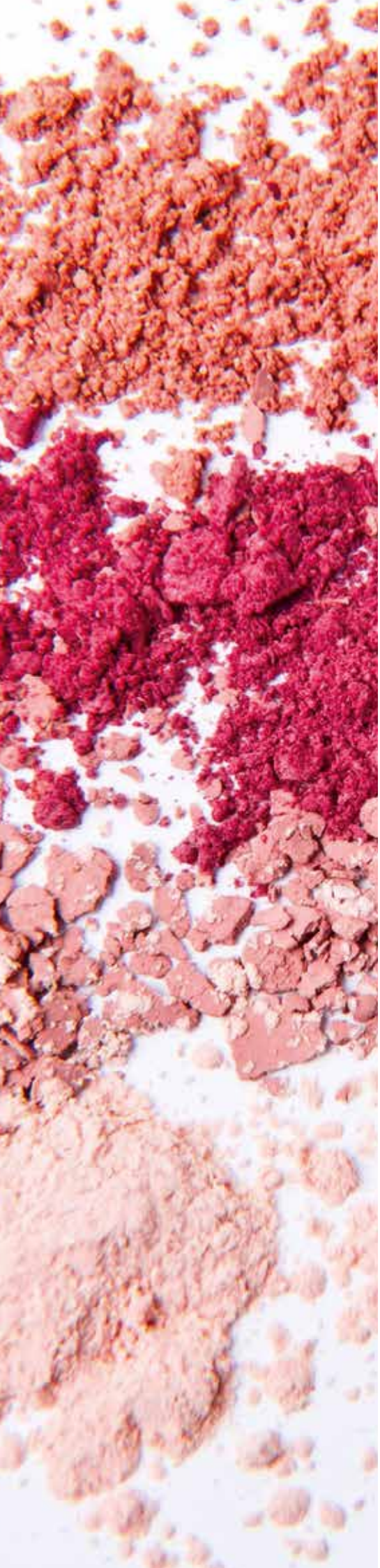
Based on preliminary analysis, the difference between predictive models developed by the team and actual observations is relatively small. This suggests that translating observations of interest into a set of properties (e.g. growth speed) that have spectral responses, could provide a new approach for data collection and understanding.

There is plenty of uncertainty regarding climate models. Through combining and linking hyperspectral imagery and analysis on a landscape scale with VNIR spectral data collected locally with the ASD FieldSpec spectroradiometer, Argonne National Laboratory's EcoSpec project is observing what is happening today at the local scale to facilitate more accurate forecasting and improved future climate models at a larger scale.



References

1) ecospec.evs.anl.gov/
2) ecospec.evs.anl.gov/data/index.cfm
3) www.evs.anl.gov/news/2015/03-02-ecospec-sensor-award.cfm



MANUFACTURERS CHOOSE **ACCURATE AND EFFICIENT PARTICLE SIZE ANALYSIS** OF POWDERS

Metals And Additives LLC (MAC) are always looking to expand their capabilities and applications so that their products can benefit their customers in a wide array of industries, even beyond plastics and batteries. Particle size analysis is a key part of the testing of their materials and products. In 2016, they purchased the Mastersizer 3000, from Malvern Panalytical. Justin Hoover, Quality Manager at MAC explains the decision-making process for the new system, the benefits of the Mastersizer 3000 and the company's experience of working with Malvern Panalytical.

The majority of the products MAC manufactures are dry powders and, consequently, the company requires flexibility to move away from wet analysis as the standard method. Though MAC had used another vendor's laser diffraction instrument without the dry dispersion module in the past, they really needed an instrument that could perform analysis of particle size distribution on both wet and dry samples.

"Many of our products will agglomerate in the liquids used for analysis," says Justin Hoover. "Some are soluble in water, causing compatibility issues with some of the liquids that could be used. We also had problems resolving issues with air bubbles during analysis and the methods were time-consuming, prone to complications and error. Solving this challenge was our key objective."

"We initially considered purchasing the module for our existing instrument to allow for dry dispersion particle size distribution analysis. However, the cost of adding capabilities, age of the instrument and our overall satisfaction with it meant that we decided to look at other instruments. As we test both dry powders and material suspended in liquid, we needed an instrument that could be versatile, as well as accurate and efficient for the analysis of particle size distribution."

In order to assess the range of instruments available, selection criteria were identified, and a number of vendors were considered. "We compared five instruments for this purchase; our criteria being accuracy, precision, ease of use, report customization, cost of software updates, instrument footprint and instrument cost." It was both Malvern Panalytical's instrument performance and the sales process that set it apart from other vendors.

"The Mastersizer 3000 exceeded the competition in almost every category for the criteria used," continues Justin. "It has the smallest footprint, is the easiest instrument to switch between wet and dry modules and we have found it to have the most user-friendly software. Other major benefits include free software updates, almost infinite report customization, the most aesthetically pleasing software and hardware; it's the easiest instrument to maintain, provides high precision results and has, overall, the best price for its class. Malvern Panalytical's approach to the sales process was also one of the best, particularly the after-sales support."

Taking a holistic view of the results shows that the technology advances made possible through the Mastersizer 3000 have been a game changer for MAC. "We've been using the Mastersizer 3000 for almost a year and our throughput has increased more than we could have imagined. The repeatability is such that multiple analyses overlaid appear as one distribution, and the software is extremely robust. Having access to the continuous software updates also adds practical functionality."

"The design of the wet dispersion module had eliminated our issues with bubbles and the dry dispersion module allows for extremely fast analysis of our dry powders. We've been very impressed with the reports generated, especially as we can include our logos and customize these for the applications for which they will be presented."

"The small footprint gives us more bench space, which is always a scarce commodity in the lab. Now, we can acquire particle size distribution on any of our products, including the soluble and difficult materials. The Mastersizer 3000 requires low and easy maintenance; we've not experienced any issues. This instrument is a dream for particle size analysis," Justin concludes.

"We've been using the Mastersizer 3000 for almost a year and our throughput has increased more than we could have imagined."

Justin Hoover
Quality Manager at MAC

METALS AND ADDITIVES LLC

addenda OMNI OXIDE POLYMER ADDITIVES GROUP

Metals And Additives LLC (MAC) is a private holding company for three separate corporations – OMNI Oxide, Addenda LLC and PAG Holdings. The headquarters for MAC is in Indianapolis, Indiana USA with manufacturing in Brazil, Indiana. All MAC locations are ISO 9001:2000 Certified. The company is a leading manufacturer and supplier of powder additives for the battery and plastics industries.

THE HISTORY OF **X-RAY TECHNOLOGY**

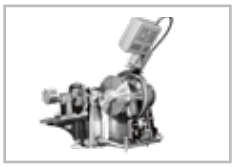
1972 - 2002

A SIX-PART SERIES

In the 1950s and 60s, X-ray diffraction (XRD) and X-ray fluorescence (XRF) became mature analytical techniques that were used in scientific and industrial laboratories all over the world. Based on the great success, Philips decided to establish a dedicated analytical X-ray business unit within the electronics concern, next to the medical and industrial X-ray operations

1895 - 1917

1917 - 1945



1945- 1972

1972 - 2002

2002 - 2017

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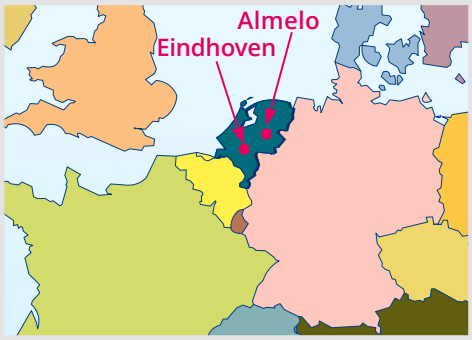
2017 - ...

PHILIPS ANALYTICAL X-RAY: TOWARDS A GLOBAL COMPANY

Almelo and Eindhoven



After several years of successfully developing and manufacturing XRD and XRF equipment in Mount Vernon, NY, USA, Philips opened a second factory for analytical X-ray products in Eindhoven, the Netherlands in 1952. For further global expansion of the business, Philips decided to establish just one business center for a worldwide supply. They chose Almelo, an industrial town in the Eastern part of the Netherlands. Here, a good industrial infrastructure and well-educated, skilled technicians were available as a previously existing textile industry in the area had been relocated to lower-wage countries.

Starting in 1961, Philips gradually moved their production, development and marketing departments to Almelo, completing the relocation in 1975 with the opening of the new application laboratories. The company became known as Philips Analytical.

Since 1918, all Philips X-ray tubes (for medical, analytical and industrial applications) had been produced in the same laboratory. To stimulate further growth of the analytical X-ray business, a dedicated factory for the development and production of analytical X-ray tubes was established in Eindhoven in 1972.

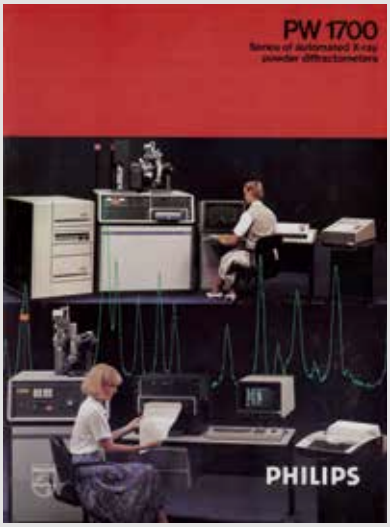
The Automated Powder Diffractometer

At the beginning of the 1970s, around 1000 X-ray diffractometers were in operation worldwide. Although most of these installations

were used for the daily analysis of powders, working with a diffractometer was far from a routine job. Only a few of the powder diffractometers were automated and X-ray diffraction was considered an academic tool.

In 1972, introduction of the Philips PW1700 APD (Automated Powder Diffractometer), known in the USA as the APD 3500, made X-ray diffraction analysis available for routine users. The new system was equipped with a software-controlled stepping motor and a microprocessor. It was operated via a teletypewriter and the additional sample changer facilitated overnight measurements on multiple samples.

The next generation of diffractometers, the Philips X'Pert line, was introduced in 1992. These instruments were equipped with optical encoders on the goniometer axes, eliminating known errors, such as backlash. A diffractionist's dream come true!

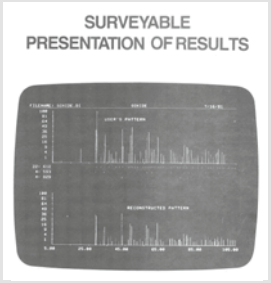


Towards automated XRD phase identification

Nowadays, phase analysis from X-ray diffraction data has become an indispensable tool for many industries and research laboratories to understand their processes. The first steps were made in the 1960s, when computer systems were introduced in the analytical laboratories, facilitating phase identification using automated search-match algorithms.

Already in 1983, Philips Analytical released APD 1700, its first complete software suite for XRD analysis. The software was designed for use with VAX and PDP computers and was later transferred to more modern computer platforms. In 1989, PC APD (automated powder diffractometry) was introduced for IBM PCs. With the launch of the X'Pert software suite in 1997, all analytical packages became available for Windows platforms.

The introduction of X'Pert HighScore in 2001 made phase identification available for both novice and experienced users. It was the start of the development of modern X-ray analytical software.



Simultaneous and sequential spectrometry

In the field of X-ray fluorescence spectrometry, automated analysis became commercially available in 1954 when Philips entered the market for X-ray analysis equipment for process and product control, introducing the Autrometer, the world's first automated spectrometer. Continuous development of the goniometer, the heart of the instrument, and the addition of a minicomputer resulted in the introduction of the PW1450 series of fully automated wavelength dispersive spectrometers around 1975. In those days, the system was also known as the Automated X-ray Spectrometer system (AXS).

Soon after, in 1979, Philips expanded its product portfolio of analytical X-ray equipment with the PW1600 series of microprocessor-based multi-channel XRF spectrometers. The simultaneous measurements of up to 28 elements made multi-element analysis extremely fast and the system was mainly used for the analysis of metals in large-scale process environments. One of the new features was 'telephone servicing' via a modem with a central location, an early form of Remote Diagnostics.

Increased speed and resolution

In the 20th century, XRD was mainly used for research because each measurement could take more than a day. This changed in 2001, when Philips Analytical introduced their revolutionary X'Celerator fast X-ray detector. Based on Real-Time Multiple Strip technology, this detector made it possible to measure up to 100 times faster than with conventional detectors without any sacrifice on data quality. High-resolution powder XRD measurements that used to take several hours could now be done in minutes. This enormous time gain paved the way for automated Rietveld analysis in the cement industry.

TARGETED TO MEET **CUSTOMERS' NEEDS**: MALVERN PANALYTICAL'S NEW APPLICATION EXCELLENCE (X) LAB IN SHANGHAI

This September, we opened a new applications laboratory in Shanghai. XPress took the opportunity to talk to Roger Liang, General Manager of Malvern Panalytical China, about how he and his colleagues are working to help our customers find the right solutions for their analytical challenges.

Roger, tell us about your background.
I graduated from China's Tsinghua University with a Mechanical Engineering degree, 27 years ago. I started my professional career as a Technical Support and Service Engineer in an international instrument company, quickly switched to Sales, and later to Business Management. I've worked in several multinational companies in a variety of industries, and the East-West differences in management style never cease to fascinate me - one of my personal challenges is combining Western management theories and experiences with Chinese culture.

When did you join Malvern Panalytical?
I joined Malvern Instruments as General Manager for China in April 2016 and was appointed as General Manager of Malvern Panalytical China in 2017. I have overall responsibility for Malvern Panalytical in China, and for our local subsidiary OMEC in Zhuhai.

I am also the Ethics Officer for Malvern Panalytical in China, ensuring that our company is always represented in a professional and ethical manner. I aim to provide strong and dynamic leadership with a clear vision for the future that empowers staff to deliver best-in-class quality in processes and services to our customers.

How is Malvern Panalytical organized in China?
In total we have 270 employees who, together with local channel partners, are responsible for the sales, marketing and support of all Malvern Panalytical products within China. Our HQ office is in Shanghai, and we have five branch offices in Beijing, Guangzhou, Chengdu, Wuhan, Xi'an and Shenyang. In Shanghai, we have a fully-equipped applications laboratory to provide pre- & post-sales application support to our customers. Additionally, we have two 'mini labs' in Beijing and Guangzhou with selected demonstration systems.

Can you tell us a little more about your new applications lab in Shanghai?

The new Application Excellence (X) Lab in Shanghai is a milestone for Malvern Panalytical China. It is convenient, efficient and targeted to meet our customers' service and support needs. It also serves as an 'Excellent Customer Service Center', where customers can communicate directly with Application Specialists to collaborate on method and solution development and performance verification, in a first-class hardware and software environment.

It is Malvern Panalytical's philosophy that although our customers share many similarities, each has a unique challenge. In our new lab, we address these challenges individually to find the best-fit solution for each customer. This partnership does not stop at the point of sale, as we view each customer project as a continuous engagement to assist and support where needed.

A good example of this is our close cooperation with a Chinese innovation company to support their work in drug development. This resulted in a joint project on the application of an automated imaging-based method for the characterization of particles in a generic semi-solid topical product (a poster was published at the American Association of Pharmaceutical Scientists (AAPS) Annual Meeting in November 2017).

Are there particular applications or markets which are key for Malvern Panalytical China?

Environmental protection is a hot topic in China, with our government showing a keen awareness of the issues of air pollution and the associated health hazards. Malvern Panalytical has been instrumental in developing soil and air pollution analysis methods, and has completed an evaluation for the Chinese Norm (HJ 830-2017 and HJ 829-2017) at the Chinese Ministry of Environmental Protection. We are also working with a number of researchers to address air pollution and soil contamination.

The **pharmaceutical industry** is another of our focus areas - we are very proud of the role we play in helping ensure the safety and efficacy of drugs. We work very closely with a number of leading pharmaceutical companies - one of them, for example, uses its 9 Mastersizer systems and 3 XRD systems to control and optimize development and manufacture of its generic drug products.

Last - but not least - **the battery market** is expected to experience double digit growth by 2020. On the streets of China, almost all small motorcycles (scooters) are now powered by electricity, and Tesla has announced the imminent construction of a mega factory in the Shanghai area. The automotive industry will account for 40% of the total demand on batteries by 2020. China is the biggest single market and we expect 40% growth in Li-ion battery cell production in 2018, meaning that there is an enormous need for expertise in production and quality control. We have a variety of solutions in our portfolio that can improve the development, production and manufacturing of batteries ***(see our overview on page 16-17)***.

How do you see the future for Malvern Panalytical in China?

China is an important market for Malvern Panalytical, as it is for many other companies, and we are continuing to invest here. We are committed to always doing business in the right way, so we work in full transparency with the Chinese government.

We continue to make our mark as experts in the development of analytical solutions and services which are tailored to address specific challenges in a variety of industries. Our new Application Excellence Lab is a clear example of our ambition and dedication to the Chinese market and we are happy to welcome you there to see how we can support you with your analytical challenges, both now and in the future.

"We are exceptionally proud of the wealth of knowledge and expertise of the Malvern Panalytical China team. Combined with our flexible portfolio of solutions, this places Malvern Panalytical in a unique position to help each customer with his or her specific challenges."

Roger Liang
General Manager
Malvern Panalytical China

WATERS AND MALVERN PANALYTICAL **COLLABORATE** TO **PROVIDE FASTER** AND **MORE DETAILED** POLYMER ANALYSIS

The emergence of new and increasingly complex polymers with large structural and compositional diversity has been a driving force in the development of advanced analytical and separation technologies for polymer characterization. Gel Permeation Chromatography has always been the technique of choice for high-resolution, size-based characterization of polymers, but now Advanced Polymer Chromatography combined with advanced detection offers faster and more detailed polymer analysis.

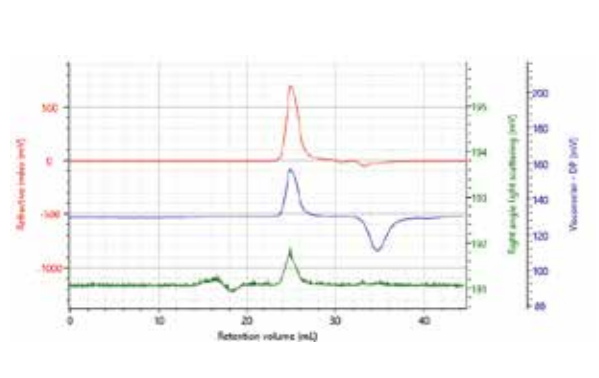
Ultra-performance liquid chromatography (UPLC), as applied by the Waters ACQUITY Advanced Polymer Characterization (APC™) system, combines high-efficiency columns with low overall system dispersion to significantly improve peak resolution, especially for low molecular weight oligomers. In addition, run times of the APC are often five times faster than with traditional Gel Permeation Chromatography (GPC), enabling higher sample throughput and more rapid method development.

In the past, it was not possible to pair this system with advanced online detectors, such as light scattering, due to limitations in their dispersion characteristics. That has now changed with Malvern Panalytical's OMNISEC REVEAL, which is optimized for use under UPLC conditions. Coupling the APC System to the REVEAL multi-detector module allows absolute molecular weight, intrinsic viscosity and hydrodynamic radius to be determined. These parameters can be used to predict polymer behaviors in solutions/ product matrices and to give a more comprehensive understanding of polymer structure. The speed of analysis and improved access to more detailed polymer information provides a more efficient pathway for the development of successful, high-quality products.

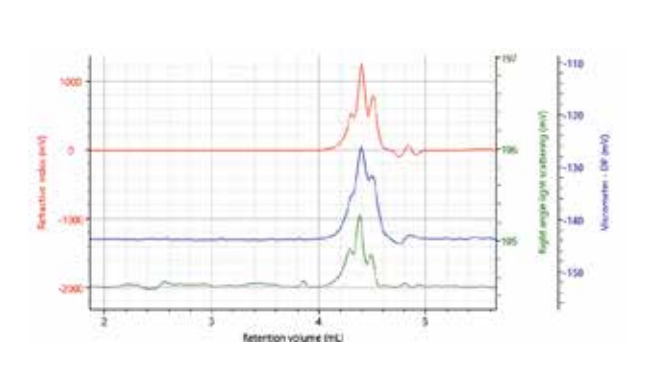
The chromatograms below show triple detection data for the same sample of a PAO separated using a standard OMNISEC GPC system and an APC-REVEAL system (ACQUITY APC pump connected to an optimized Malvern Panalytical OMNISEC REVEAL).

The APC is able to better resolve different macromolecular components, which in turn provides a more complete characterization of the sample and better understanding of any underlying changes that might cause differences in performance. Combining the ACQUITY APC with the OMNISEC REVEAL therefore gives the best of both worlds - faster and more detailed polymer analysis.

In addition, the greatly reduced quantity of solvent and time required to obtain comprehensive characterization of samples using the APC-REVEAL system presents obvious benefits to both researchers and manufacturers working



Triple detector chromatogram of a 6 cP poly-α-olefin (PAO) separated using a standard OMNISEC GPC system showing refractive index (red), right angle light scattering (green) and viscometer (blue) signals. The single peak had a measured molecular weight of just 800 Da.



Triple detector chromatogram of a 6 cP poly-α-olefin (PAO) separated using the combined APC-REVEAL system showing refractive index (red), right angle light scattering (green) and viscometer (blue) signals. The PAO sample is now partially resolved into its 3 component parts of 420 Da, 70 Da and 1160 Da, in proportions of 33%, 51% and 16% respectively.

with polymeric materials. In the example on page 14, the use of a combined APC-REVEAL system represents a saving both in solvent consumption (in this case, ~30 mL) and time for analysis (in this case, ~35 mins) per analysis.

"Malvern Panalytical is proud to work with Waters to leverage the high sensitivity and low dispersion characteristics of the OMNISEC REVEAL advanced detector module, which are ideally suited to the APC system's high-resolution and high-efficiency separation characteristics. We believe that researchers will be delighted with the analytical abilities of the combined system, which offers extraordinary performance for the analysis of both natural and synthetic polymers and provides visibility of details that would otherwise be missed.

The combination of Waters' AQUITY APC and Malvern Panalytical's OMNISEC REVEAL opens new doors in polymer research and development which will translate directly to better product performance," stated Steven Horder, Vice President, Advanced Materials, Malvern Panalytical.

Waters ACQUITY UPLC system (left) and Malvern Panalytical OMNISEC REVEAL

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Founded by Jim Waters in 1958, Waters serves life, materials and food sciences through a connected portfolio of chromatography, mass spectrometry, and thermal analysis innovations.

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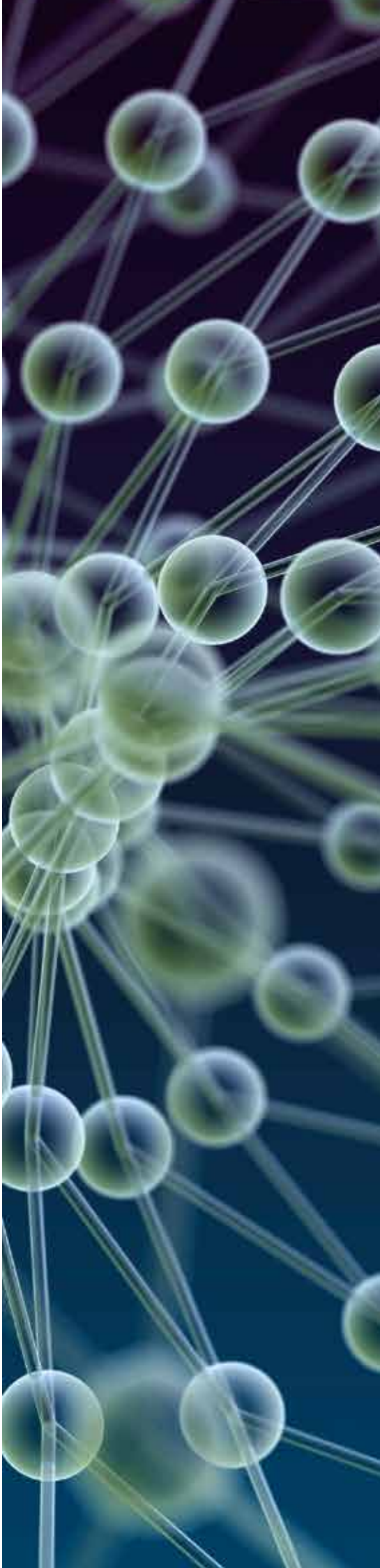
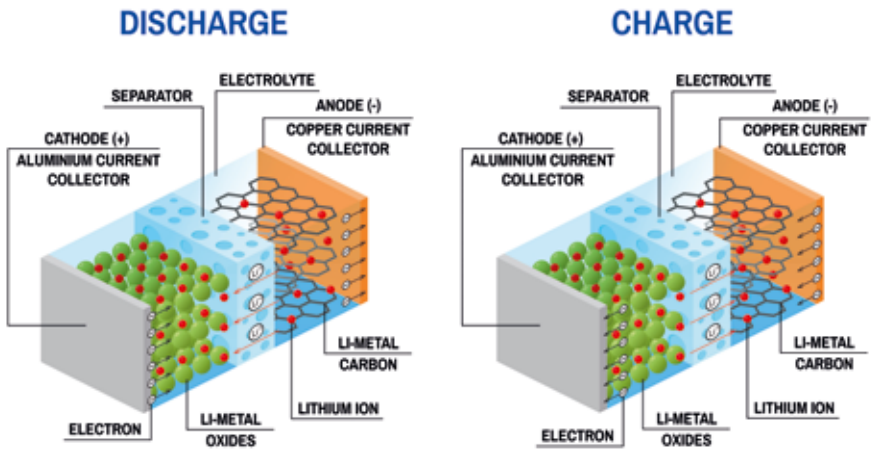


TOOLS FOR A COMPREHENSIVE **CHARACTERIZATION OF LI-ION BATTERIES**

The increased demand for portable electronic devices, including mobile phones, laptops and new 'wearables', has required advances in battery technology to provide a low-cost, lightweight, long-lasting and stable power source. With fossil fuels dwindling and CO_2 regulations becoming more stringent, battery technology is increasingly being used in applications such as renewable energy storage and electric vehicles, which require ever more lightweight, safe, high-power and fast-charging batteries.



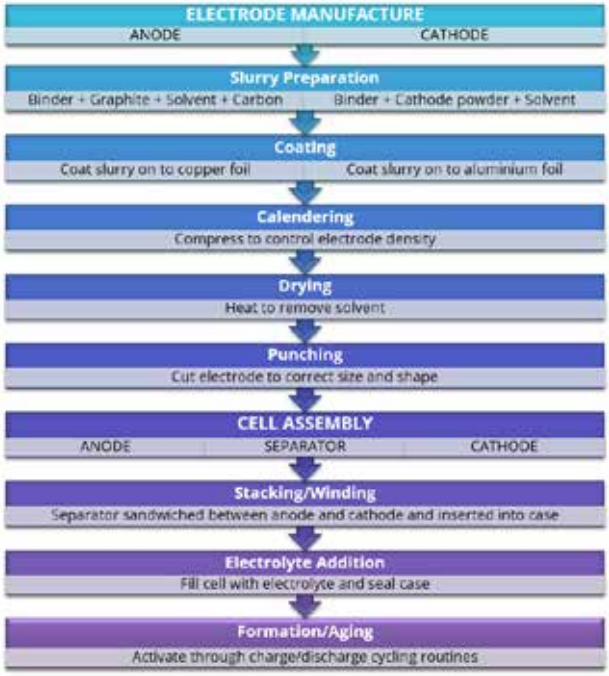
Schematic of a Li-ion battery. During discharge, lithium ions (Li^+) move from the anode to the cathode, conducted via the electrolyte, with flow in the reverse direction occurring when the battery is charged. Anodes are typically graphite-based, while cathodes are often manufactured using lithium-iron phosphate, lithium-cobalt oxide, lithium-manganese oxide, lithium-nickel cobalt manganese oxide, etc.

The cornerstones of battery performance are power, which impacts current and discharge characteristics, and energy storage capacity. Battery power is determined by the rate of reaction between the electrodes and the electrolyte, while storage capacity is a function of the volume of electrolyte within the cell. These properties are intrinsically linked to the intercalation structure and primary particle size of the electrode particles, which determine how well the mobile ions are taken up and released by the electrode[1]. Particle size distribution and particle shape also influence particle packing and hence the volume of electrolyte that can be accommodated within the interstitial voids of the electrode, which affects storage capacity.

Particle sizing of electrode materials is commonly performed using laser diffraction technology, such as the Mastersizer 3000; although the Zetasizer Nano can also be employed for analyzing the much smaller particles used for electrode materials or separators. **Automated imaging** (Morphologi 4) is commonly employed for particle shape analysis but can also be coupled with Raman spectroscopy to give particle-specific structural information. The primary tool for **studying the structure of electrode materials**, however, is X-ray diffraction (XRD) with Aeris or Empyrean diffractometers widely used in this sector. Empyrean also allows small-angle X-ray scattering (SAXS), an important tool for **evaluating the primary particle size or crystallite size of nanoparticle electrodes** which tend to exist in aggregated form.

Manufacture of the cathodes and anodes involves mixing the active electrode material with some form of conductive additive such as carbon (e.g. carbon black and/or graphite) and a polymeric binder dissolved in a solvent to form a slurry. The slurry/suspension is then applied to a metal foil in a continuous coating process and the solvent driven off to produce a dried coating which is subjected to calendaring, a compression process that involves feeding the coated foil through a series of rollers. The electrodes are then punched or cut to size prior to winding or stacking between the separator film before inserting into the case, wetting with electrolyte, and sealing. The cells are then activated over several weeks through a series of charge/discharge cycles.

These processes exert a significant influence on electrode structure and are related to the rheological, or flow properties, of the battery slurry. These properties can be influenced by the extent to which raw materials are dispersed during the slurry manufacturing process, affecting the size of particles deposited on the foil or the impact of compressive forces applied during calendaring, which affects the porosity of the finished coating. These are not discrete effects - rather changes made during one step can have an impact on consecutive processes, as well as the properties of the finished electrode[2].



The manufacture of Li-ion battery electrodes is a multi-step process with each step having potential to influence the electrochemical performance of the finished device[2]

Slurry rheology is influenced by the size, shape, concentration and charge of dispersed components, hence it is important to optimize electrode particle properties not just for their individual electrochemical performance but for the electrode manufacturing process as a whole[3]. Ultimately it is the performance, and safety, of the final battery cell which is of critical importance. The most common tool for **measuring the general rheology of battery slurries** is a rotational rheometer, such as Kinexus, although a capillary rheometer such as the Rosand RH7 or RH10 may better represent the electrode coating process[4].

So, what happens to the electrode structure during initial formation, as well as the repeated charge/discharge cycling of the fabricated battery cell? The repeated transfer of charge-carrying ions in and out of the electrode lattice puts strain on the cell structure and can induce unwanted phase changes, even more so if fast charging and/or discharging is required. Deterioration in performance is most commonly due to the build-up of defects within the electrode which hinders ion mobility. **The mechanism of these processes on the electrode structure** can be studied with *in operando* XRD, using hard X-rays (Mo or Ag Kα) capable of penetrating a battery cell, ideally (but not limited to) a pouch cell[5].

It is then possible to correlate variations in the crystallographic structure of the electrodes with the amount of Li incorporated in to them. The spatial resolution of *in operando* transmission XRD (approx. 1 mm) also makes it an ideal tool for **probing localized wear**, since cells close to the connector tend to degrade faster than those further away. It can also **identify areas** within the cell that are not functioning properly due to ageing or incomplete wetting with electrolyte. Empyrean equipped with the GaliPIX^{3D} detector is ideally suited for this application since it offers the highest resolution data in the shortest possible time frame.

References

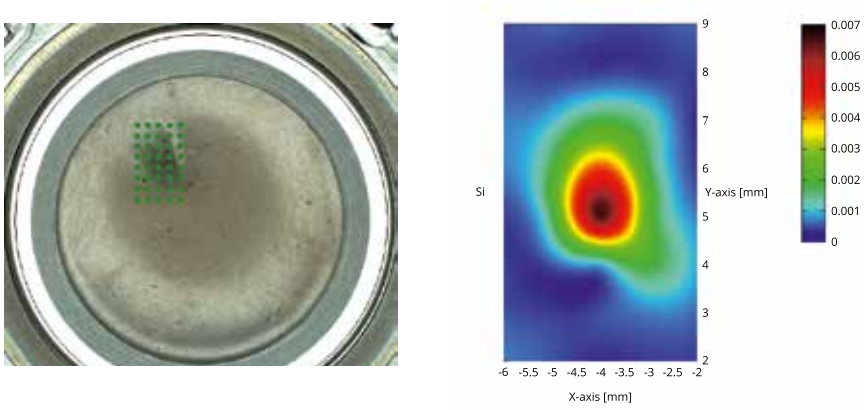
1) Julien, C.M., Mauger, A., Vijh, A., Zaghib, K.; Lithium Batteries: *Science and Technology*; Springer: Heidelberg, Germany, 2015
2) Bockholt, H. *et al*. 'The interaction of consecutive process steps in the manufacture of lithium-ion battery electrodes with regard to structural and electrochemical properties'; *Journal of Power Sources* 325 (2016) 140-151
3) White paper: Exploring the impact of particle characteristics on suspension rheology
4) White paper: Establishing an analytical toolkit for the optimization of Li-ion battery electrode manufacturing
5) Application note: High-quality *in operando* X-ray diffraction analysis of pouch bag lithium-ion batteries

REDUCED ANALYSIS TIMES FOR INCLUSIONS AND ELEMENTAL MAPPING

Most producers strive to deliver a homogeneous product where any imperfection or inclusion is one too many. Think of a polymer containing inclusions or the presence of unexpected and possibly harmful or costly elements in your product. But how do you find out what kind of irregularity is present and why? How can you improve your product quality and resolve any production issues? HiPer Small Spot Mapping (HiPer SSM) with a Zetium spectrometer could be the solution for you. It only requires minimal sample preparation and delivers results in a much shorter time than alternative techniques.

Malvern Panalytical's renowned Zetium X-ray fluorescence (XRF) spectrometer not only offers the possibility to perform bulk analysis but recently our developers have added the new HiPer SSM option to the instrument's versatile SumXcore platform. This option can help you to perform inclusion analysis in just a few minutes or map more than 100 mm^2 in only a few hours, reducing measurement times by up to 80%.

It covers almost the entire periodic table of the elements from sodium to americium, so that no unexpected element remains undetected.



Small spot mapping of a dark spot or defect in a polyolefinic sample reveals an accumulation of silicon in the measured area as depicted in the photograph on the left.

The method preserves your samples and can be scheduled to run over the weekend or overnight. Zetium's SumXcore platform is a unique combination of wavelength dispersive (WD) and energy dispersive (ED) technologies, combining their power: HiPer SSM uses the ED core to provide fast simultaneous and multi-element analysis on small spots with the uncompromised accuracy and precision of the WD core for bulk analysis. It offers a unique possibility to reveal local details in your sample for quality checks or the detailed examination of a reaction mechanism. Your local sales team can inform you about upgrade possibilities for your existing small spot mapping option.

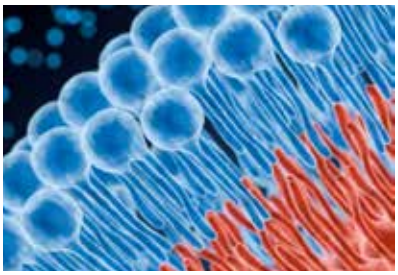
"The HiPer Small Spot Mapping option brings further diagnostic capabilities to the Zetium's bulk analysis role in process and quality control."

Simon Milner
Director of Elemental Analysis at Malvern Panalytical

COMPLEMENTARY TECHNIQUES FOR **THOROUGH CHARACTERIZATION OF LIPOSOMES**

Liposomes have been attractive drug delivery systems for decades due to their biological lipid composition and their structural resemblance to cell membranes. This suggests a high potential for metabolic compatibility and biocompatibility, and low potential for toxicity or immunogenicity. Liposomes comprise spherical vesicles with an aqueous core enclosed by one or more phospholipid bilayers or lamellae, and are frequently classified based on their size, polydispersity, and number of bilayers.

Control over these parameters has remained a challenge for most preparation methods and is further accentuated when moving from a laboratory to industrial scale. Comprehensive biophysical characterization of liposome systems is critical to understanding and optimizing their fabrication and function.



In a recently published whitepaper, we present a cost-effective analytical suite for direct characterization of liposomal critical parameters. **Nanoparticle tracking analysis (NTA)** by the NanoSight range measures size, concentration and fluorescence. **Dynamic (DLS) and electrophoretic (ELS) light scattering** by the Zetasizer product range measures size, zeta potential, and payload stability. Small- and wide-angle X-ray scattering (SAXS/WAXS) measurements enable the determination of lamellarity, structure and dimensions of the lipid bilayers.

The whitepaper can be found on **www.malvernpanalytical.com/liposomesWP**

ENSURE THE SAFETY OF YOUR PHARMACEUTICAL PRODUCTS

Elemental impurities in pharmaceuticals may come from catalysts, ingredients or production vessels. As they do not provide any therapeutic benefit to the patient and may even be hazardous, assurance of USP <232> compliance within levels is required. Frequent and fast elemental analysis during production is key to reliable accurate quality control.

USP chapter <735> and EP 2.2.37 describe the use of X-ray fluorescence (XRF) spectrometry for pharmaceutical elemental impurity analysis. Malvern Panalytical has developed a unique solution for impurity control, consisting of the Epsilon 4 XRF benchtop instrument and three sets of dedicated pharmaceutical impurity standards. Contact your local sales organization to learn more about Malvern Panalytical's USP-compliant analysis solution to monitor elemental impurities in your pharmaceutical products.

COLOPHON

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

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