OCTOBER 2021, ISSUE 100

WELCOME

October is a great month to celebrate advances in science and technology. With the announcement of the Nobel Prizes, it's always a good time to reflect on one's contributions to the advancement of humanity through efforts to improve lives through research. In this issue we illustrate the wide range of technology interests that define Rigaku as a company. From the application of small angle X-ray scattering to real-space modeling for complex structures to furthering development of new batteries through *operando* XRD measurements. From improved measurements of sulfur in ultra-low sulfur diesel (ULSD) through EDXRF to identification of fentanyl precursors using handheld Raman spectrometers. Contents this month covering WDXRF range from the qualitative analysis of aluminum alloys to the use of the technique in the pharmaceutical market.

PUBLISHED RESEARCH FROM RIGAKU'S X-RAY RESEARCH LABORATORY

Real-space Modeling for Complex Structures Based on Small-angle X-ray Scattering

Kazuhiko Omote and Tomoyuki Iwata, *Rigaku Corporation*
J. Appl. Cryst. (2021). **54**, 1290–1297

A three-dimensional real-space model has been created for hierarchical materials by matching observed and simulated small-angle X-ray scattering patterns. The simulation is performed by arranging the positions of small primary particles and constructing an aggregate structure in a finite-sized cell. In order to avoid the effect of the finite size of the cell, the cell size is extended to infinity by introducing an asymptotic form of the long-range correlations among the primary particles. As a result, simulations for small-angle X-ray scattering patterns can be performed correctly in the low-wavenumber regime ($<0.1\ nm^{-1}$), allowing the model to handle hundred-nanometre-scale structures composed of primary particles of a few nanometres in size. An aerogel structure was determined using this model, resulting in an excellent match with the experimental scattering pattern. The resultant three-dimensional model can generate cross-sectional images similar to those obtained by transmission electron microscopy, and the calculated pore-size distribution is in accord with that derived from the gas adsorption method.

Click here to read the paper.

FEATURED APPLICATION NOTES

XRD

Operando Measurement of Laminated Lithium Ion Battery Using 2D Detector

Rigaku Corporation

To develop lithium ion secondary batteries with high capacity, high reliability, and long life, it is essential to evaluate the stability of the electrode materials during the charge/discharge process. The laminate cell attachment enables the reproduction of a high-speed charge/discharge process while keeping the sample temperature constant, and simultaneously allows the collection of transmission X-ray diffraction images. Using this attachment with an X-ray diffractometer equipped with a Mo X-ray source and a 2D detector collecting up to 131 diffraction images per second, it is possible to observe the rapid phase transition occurring inside the battery cell.
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XRD

Operando Transmission XRD Measurement of All-solid-state Lithium-ion Battery Using Ag Source

Rigaku Corporation

All-solid-state batteries that use solid electrolytes are superior to existing lithium ion batteries that use liquid electrolytes in terms of safety, higher capacity, and reduced size. Battery performance is closely related to changes in the crystal phase of the positive electrode material during charge and discharge cycles; therefore, operando measurements using short-wavelength X-rays with high penetration power are being actively conducted. In addition to Mo radiation, Ag radiation can be used as a short wavelength X-ray source. By combining Ag radiation and convergent beam optics, operando measurements can be performed by transmission geometry even for all-solid-state batteries where sample absorption is significant.
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EDXRF

Analysis of Ultra-low Sulfur Diesel

Applied Rigaku Technologies

Regulations around the world have limited the amount of sulfur in various fuels oils. In some regions, sulfur in diesel is limited to 10–15 ppm, while other areas of the world are working to bring sulfur levels down to 50 ppm and lower. Aside from automobile, truck, and some bunker fuels, ULSD is also mandated for use as a starter fuel or for backup electricity generation in coal-fired power plants and nuclear power plants in some regions. A fast, reliable method of measuring and monitoring sulfur concentration throughout the petroleum industry is vital.

The application note demonstrates the performance for the measurement of sulfur in ULSD (ultra-low sulfur diesel) using the Rigaku NEX CG II monochromatic indirect excitation EDXRF analyzer with polarization.
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Raman

Identification of Fentanyl Precursors with Handheld 1064 nm Raman

Rigaku Analytical Devices

Transnational Organized Criminal (TOC) groups and their proxies have expanded their illicit drug production capabilities to include the deadly synthetic opioid, fentanyl. Fentanyl has become the drug production of choice among criminal cartels because it is 50 times more addictive than heroin, it can be produced using a small number of precursors, and requires less time and space to manufacture.

The Rigaku series of handheld 1064 nm Raman spectrometers provide a nondestructive, reliable and safe method for analyzing and identifying dangerous chemicals such as fentanyl. More importantly, the identification of mislabeled fentanyl precursors has become a priority for law enforcement and regulatory agencies in their attempts to disrupt the PCSC networks and street-level drug distribution. Rigaku handheld Raman can scan through translucent packaging, greatly reducing the risk of exposure to the officer.
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WDXRF

Quantitative Analysis of Aluminum Alloy on Supermini200

Rigaku Corporation

Aluminum alloy, which has the valuable properties of being both "light" and "strong", is used in many industries such as automobile and aircraft. Since aluminum alloy has a broad range of grades whose characteristics are strongly dependent on the elemental compositions, it is very important to control the components. X-ray fluorescence (XRF) analysis quickly and easily offers precise elemental analysis results to make con- trol of the components in aluminum alloy possible. This application note demonstrates the excellent performance of Supermini200 in aluminum alloy analysis.
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WEBCAST SUMMARY

Direct CNO-X Analysis of Solid Forms

The pharmaceutical industry is a fast-paced environment, requiring frequent sample testing throughout development and manufacturing to ensure product purity and quality. In a setting where speed is crucial, employing analytical methods that require minimal sample preparation while offering rapid analyses is highly advantageous. This is readily achieved through analysis of solids via X-ray fluorescence spectroscopy (XRF), which can provide the elemental composition of solids measured directly, with minimal to no sample preparation. The primary elements of interest for a particular pharmaceutical application are carbon, nitrogen, and oxygen (CNO), all of which can be quantified easily with XRF instrumentation. CNO composition is critical to the determination of stoichiometry in the solid form selection processes—salt selection and polymorph, hydrate, solvate evaluations. Elements beyond CNO can be measured by XRF systems due to the broadly applicable mechanism underlying XRF measurement technology. This makes the technology useful in many different pharmaceutical applications, such as analysis of catalyst residues in APIs and intermediates, evaluation of catalysts for poison element impurities, and as a tool to analyze for blend uniformity of key excipients and certain APIs in formulations.

This webcast summary covers the basics of XRF as applied to solid analysis as well as experimental results from representative pharmaceutical samples.
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UPCOMING RIGAKU WEBINARS

Thermal Analysis Technical Seminar: Let's Evaluate Materials With EGA (STA-MS & STA-FTIR): Principles, Applications & Tips

November 25, 2021 12 AM | CDT

This webinar is a beginner's course. The presentation will focus on basic principles of evolved gas analysis (STA-MS and STA-FTIR) and the measurement conditions affecting measurement results. During the presentation, we will also highlight applications and show some videos on sample preparation and available options.
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Diffraction Methods for MOF Investigations

December 2, 2021 9 AM | CDT

In the field of Metal-Organic Framework materials, structural investigation plays the most crucial role. It is also often combined with other analytical methods to allow drawing a connection between the structure and physical properties of the framework. As a result of those multi-technique approaches, a border between a single crystal and powder diffraction techniques often disappears. In this TOPIQ webinar, we would like to present you with standard and entirely new Rigaku solutions for diffraction methods for MOF research.
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