X-ray Diffraction & Elemental Analysis

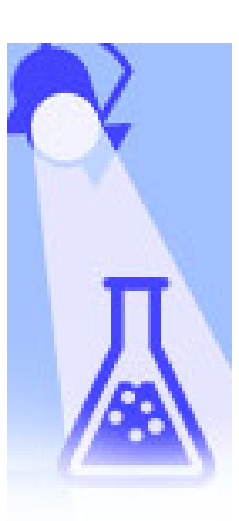
Welcome

March is always a busy conference month for Rigaku, and 2016 is no exception. Please come by and see us at the following materials science events: PITTCON, ACS – Spring, American Physical Society, Laborama, ARABLAB and MRS – Spring. We promise to have new and interesting things to discuss.

For your continuing education, we offer the sixth installment of our new series "Introduction to single crystal X-ray analysis," entitled "Refinement of disordered structure." Our featured technical paper is a primer on " X-ray Thin-film Measurement Techniques: IV. In-plane XRD measurements."

To reminisce about the work of yet another great scientist, this month's video is "Inside Einstein's Mind," from the PBS NOVA science television series. For those of us who attended high school and/or university in the 1970s, NOVA in the U.S. and Horizon on BBC2 were " must see" science television. Many legacy episodes are available on the world wide web and are worth a look … as well as perfect for sharing with children interested in STEM curricula. Check out the news and papers sections at the bottom of the page for the latest developments in materials science. Enjoy the newsletter.

R.C. Tisdale, Ph.D. – Editor



Lab in the Spotlight

The IUCr-UNESCO Open Lab Cambodia 2

The IUCr-UNESCO RigakuOpenLab Cambodia 2, was held on January 11-15, 2016, organized by the Department of Geo-resources and Geotechnical Engineering and supported by the IUCr, UNESCO and Rigaku in collaboration with the Institute of Technology of Cambodia. The initiative aimed to further disseminate and raise awareness of the importance of crystallographic education, which could benefit Cambodian society, to the public—especially to the top management of both academic and government institutions who could take part in policy-making decision. **For more >**

Introduction to Single Crystal X-ray Analysis VII

Refinement of disordered structure

Rigaku Corporation

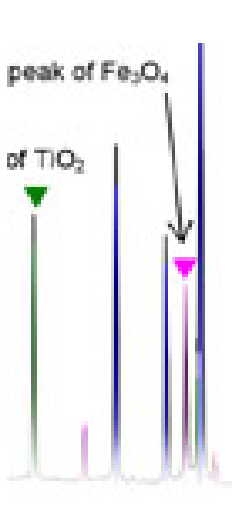
This paper starts by describing what a disordered structure is, and then introduces how to determine disordered structures, and methods of refinement using the SHELX-97 structure refinement program. **For more >**

Featured XRD Rigaku Journal Article

X-ray Thin-film Measurement Techniques IV

Rigaku Corporation

In this article, the in-plane XRD technique used for the characterizations of extremely thin films, as well as the crystallinity and orientational relationships in complex heteroepitaxial systems, is explained.
For more >

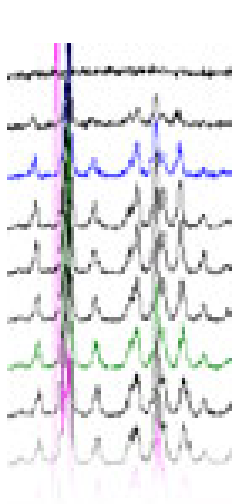


XRD Application Note

Quantitative analysis of a 4-component sample using RIR method

Rigaku Corporation

The calibration curve method is the most common method for quantitative analysis using X-ray diffractometry. However, there are cases where this method cannot be used due to problems involving obtaining the standard samples needed for plotting calibration curves, and the time needed to prepare and measure samples. In these cases, quantification using the RIR (Reference Intensity Ratio) method enables quantitative analysis without the complication of sample preparation and measurement. **For more >**

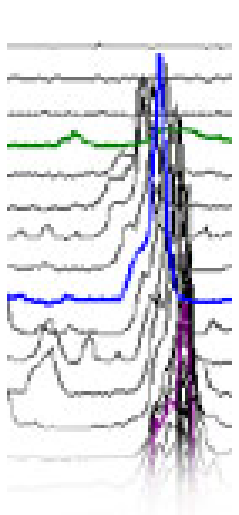


XRD Application Note

Crystallization behavior of ionic liquid at low temperatures (1)

Rigaku Corporation

Ionic liquids, consisting of molecular cations and anions, are regarded as the third most important liquid following water and organic solvents. Due to their high electrical conductivity, they have especially gained attention in relation to electrochemical applications such as fuel cells or solar cells. However, research continues, as there are still many open questions regarding the relation between their structures and their physical properties. In this Application Byte, a system featuring simultaneous XRD and DSC measurements was used to evaluate the crystal structure of ionic liquids at temperatures around -70°C. **For more >**



XRD Application Note

Crystallization behavior of ionic liquid at low temperatures (2)

Rigaku Corporation

Measurements of mixed solutions of the ionic liquid DEME (N,N-diethil-N-methyl-N-2-methoxyethylammonium tetrafluoroborate) with either 0.6 mol% or 0.9 mol% water content were performed. It has been shown that mixed solutions with low water content crystallize at low temperatures (for details, see B-XRD 1026). Using a system that supports simultaneous XRD and DSC measurements, both solutions were first cooled to below -70°C before observing the behavior of their crystalline grains when heated to room temperature. The evaluation of the crystalline grains with XRD was performed in a rocking curve measurement with 2θ fixed and only θ scanning. **For more >**

WDXRF Application Note

Analysis of S, Fe, Ni and V in Residual Oil According to IP610/13

Rigaku Corporation

Residual oil, which contains various elements such as S, Fe, Ni, V and others, is obtained after distilling crude oil and used for marine fuel or electric power plant fuel. When residual oil is burned, some elements can cause damage to marine engines and the environment. XRF is a powerful tool for analyzing these elements in residual oil. This application note demonstrates that ZSX Primus is compliant with IP610/13 for the analysis of S, Fe, Ni and V. **For more >**

EDXRF Application Note

Analysis of Metals in Aerosols on Air Filters

Applied Rigaku Technologies

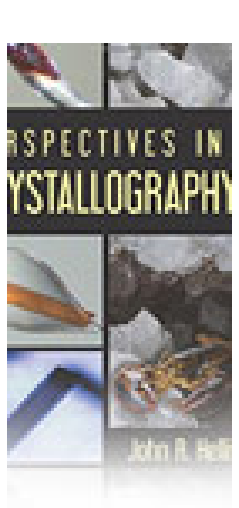
Element analysis of aerosols and particulate matter released in smokestacks and other industrial gas discharge is vital to ensure that environmentally acceptable levels of toxic and hazardous elements are released properly into the air. Monitoring smoke or other gaseous waste is important in many areas, such as industrial manufacturing, coal-fired power plants, chemical and plastics production, etc., in order to minimize air pollution and the release of toxic metals in compliance with US EPA and other world and regional or local governing regulations. As a tool to help ensure compliance and proper release, Rigaku offers the NEX DE EDXRF analyzer with 60 kV excitation source and high resolution and throughput Si Drift Detector, giving the analysts and technician alike a fast, simple, yet powerful means for monitoring elemental analysis of air filters. **For more >**

Raman Application Note

Drug Product Identification Using Handheld Raman

Rigaku Analytical Devices

Handheld Raman spectroscopy is well accepted as a technique for raw material and excipient identification within the pharmaceutical industry. Using the correct Raman excitation wavelength can extend its capabilities into the areas of drug substance and drug product identification. **For more >**

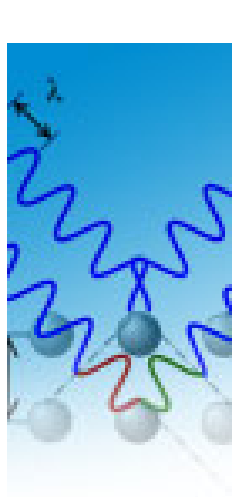


Scientific Book Review

Perspectives in Crystallography

By John Helliwell

Helliwell's *Perspectives* is a collection of articles and lectures by the author celebrating the 100th anniversary of the first crystal structures (1912) and the UNESCO International Year of Crystallography. The book covers a broad range of topics in crystallography from the author's perspective, hence the title. Some of the chapters have already been published in *Crystallography Reviews*.
For more >



Material Analysis in the News

News for February 2016

February 1, 2016. Researchers assumed that tiny objects would instantly blow up when hit by extremely intense light from the world's most powerful X-ray laser at the Department of Energy's SLAC National Accelerator Laboratory. But to their astonishment, these nanoparticles initially shrank instead — a finding that provides a glimpse of the unusual world of superheated nanomaterials that could eventually also help scientists further develop X-ray techniques for taking atomic images of individual molecules.

February 4, 2016. The Gordon and Betty Moore Foundation has awarded $13.5 million to an international collaboration led by Stanford University, to develop a prototype of an "accelerator on a chip" over the next five years. SLAC and two other national labs provide key in-kind contributions in support of this expansive university effort. Moore Foundation's funding will help scientists ideally create a complete working prototype the size of a shoebox.

February 5, 2016. David Hawthorn and his team at the University of Waterloo have used soft X-ray scattering to watch superconducting behavior in cuprates. What they found was the first direct experiment evidence of something called electronic nematicity, which occurs when electron clouds snap into an aligned and directional order, such as stripes and grids, in certain types of high-temperature superconductors.

February 9, 2016. Jüstel, Friesecke and James propose a new X-ray diffraction method for studying non-crystalline but symmetric structures, such as helices, that relies on matching the symmetry of the incoming radiation to the symmetry of the structure to be studied. Send twisted X-rays onto a helical structure, align the waves, the structure and the detector axially, and the outgoing radiation shows sharp, discrete peaks as the incoming wavelength and the amount of twist are varied. Structure prediction from the diffraction pattern then works in exactly the same way as in the case of crystals.

February 10, 2016. An international team of researchers led by X-ray scientist Christoph Bostedt of the U.S. Department of Energy's (DOE) Argonne National Laboratory and Tais Gorkhover of DOE's SLAC National Accelerator Laboratory used two special lasers to observe the dynamics of a small sample of xenon as it was heated to a plasma. The researchers used an optical laser to heat the sample cluster and an X-ray laser to probe the dynamics of the cluster as it changed over time. As the laser heated the cluster, the photons freed electrons initially bound to the atoms; however, these electrons still remained loosely bound to the cluster.

February 15, 2016. Researchers from the University of Illinois Urbana-Champaign sliced russet potatoes into 45-millimeter diameter, 1.65-millimeter thick round pieces and then fried them in soybean oil. A team analyzed how the porosity is altered when the slices are dunked, if the paths connected to the pores are affected and how much oil is absorbed. With the help of X-ray micro-computed tomography (μCT), they found the number and size of pores in the potato increased the longer it fried, allowing it to absorb more oil.

February 15, 2016. Researchers from Arizona State University (ASU), Deutsches Elektronen-Synchrotron (DESY) and Stanford Linear Accelerator Laboratory (SLAC) describe a simple way to determine the X-ray crystal structure of proteins and other molecules, many of which are inaccessible by existing methods. The work demonstrated that it is possible to use the continuous diffraction of imperfect crystals to obtain better molecular images than with Bragg peaks alone.

February 17, 2016. Astro-H is a facility-class space mission launched on a JAXA H-IIA into low Earth orbit on Feb 17 , 2016 at 5:45 pm JPS from Tanegashima Space Center in Japan. Soon after launch, ASTRO-H was named Hitomi. Hitomi is Japan's sixth X-ray astronomy mission, and was primarily developed at the Institute of Space and Astronautical Science of Japan Aerospace Exploration Agency (ISAS/JAXA). The Hitomi mission objectives are to study of the structure of the universe.

February 17, 2016. A team from Tohoku University and the University of Tokyo have found a way to make the "wonder material" graphene superconductive, which means electricity can flow through it with zero resistance. The new property adds to graphene's already impressive list of attributes, like the fact that it is stronger than steel, harder than diamond, and incredibly flexible.

February 22, 2016. Japanese researchers have succeeded in using the immensely powerful X-ray pulses from the free electron laser (XFEL) facility SACLA to investigate excited-state induced transient lattice dynamics on sub-picosecond time scales in phase-change materials via X-ray diffraction. The work revealed a previously unknown transient state, formed on picosecond time scales, that strongly suggests that the usual thermally induced nanosecond order transition of conventional phase-change memory may be used to speed up memory switching to picosecond time scales.

Recent Scientific Papers of Interest

Papers for February 2016

Recent Scientific Papers of Interest is a monthly compilation of material analysis papers appearing in recently released journals and publications. *See below*

Combined microtomography, thermal desorption spectroscopy, X-ray diffraction study of hydrogen trapping behavior in 7XXX aluminum alloys. Bhuiyan, Md. Shahnewaz; Toda, Hiroyuki; Peng, Zhang; Hang, Su; Horikawa, Keitaro; Uesugi, Kentaro; Takeuchi, Akihisa; Sakaguchi, Nobuto; Watanabe, Yoshio. *Materials Science & Engineering: A*. Feb2016, Vol. 655, p221-228. 8p. DOI: 10.1016/j.msea.2015.12.092.

Immediate screening of lead exposure in the workplace using portable X-ray fluorescence. Gorce, Jean-Philippe; Roff, Martin. *Journal of Occupational & Environmental Hygiene*. Feb2016, Vol. 13 Issue 2, p102-111. 10p. 2 Diagrams, 3 Charts, 4 Graphs. DOI: 10.1080/15459624.2015.1091959.

XRD, HRTEM, magnetic, dielectric and enhanced microwave reflection loss of $GaFeO_3$ nanoparticles encapsulated in multi-walled carbon nanotubes. Mahapatra, A.S.; Mitra, A.; Mallick, A.; Chakrabarti, P.K. *Ceramics International*. Feb2016, Vol. 42 Issue 3, p3826-3835. 10p. DOI: 10.1016/j.ceramint.2015.11.047.

***In situ* SAXS/WAXS investigation of the structural evolution of poly(vinylidene fluoride) upon uniaxial stretching.** Defebvin, Juliette; Barrau, Sophie; Stoclet, Grégory; Rochas, Cyrille; Lefebvre, Jean-Marc. *Polymer*. Feb2016, Vol. 84, p148-157. 10p. DOI: 10.1016/j.polymer.2015.12.041.

XRD and XAFS study on structure and cation valence state of layered ruthenium oxide electrodes, Li_2RuO_3 and $Li_2Mn_{0.4}Ru_{0.6}O_3$, upon electrochemical cycling. Mori, Daisuke; Kobayashi, Hironori; Okumura, Toyoki; Nitani, Hiroaki; Ogawa, Masahiro; Inaguma, Yoshiyuki. *Solid State Ionics*. Feb2016, Vol. 285, p66-74. 9p. DOI: 10.1016/j.ssi.2015.09.025.

Direct investigations of temperature related structure transitions in strained poly(butylene succinate) with SAXS and WAXS. Zhang, Wenyang; Zhou, Chengbo; Zhang, Yao; Xue, Feifei; Luo, Baojing; Li, Hongfei; Ren, Xiangkui; Li, Jingqing; Shang, Yingrui; Wu, Zhonghua; Jiang, Shichun. *Colloid & Polymer Science*. Feb2016, Vol. 294 Issue 2, p321-328. 8p. DOI: 10.1007/s00396-015-3789-z.

Chemical mapping of mine waste drill cores with laser-induced breakdown spectroscopy (LIBS) and energy dispersive X-ray fluorescence (EDXRF) for mineral resource exploration. Kuhn, Kerstin; Meima, Jeannet A.; Rammlmair, Dieter; Ohlendorf, Christian. *Journal for Geochemical Exploration*. Feb2016, Vol. 161, p72-84. 13p. DOI: 10.1016/j.gexplo.2015.11.005.

XRD-TEM-AEM comparative study of *n*-alkylammonium smectites and interstratified minerals in shallow-diagenetic carbonate sediments of the Basque-Cantabrian Basin. Nieto, Fernando; Arroyo, Xabier; Aróstegui, Javier. *American Mineralogist*. 2016, Vol. 101 Issue 2, p385-398. 14p. DOI: 10.2138/am-2016-5301.

Monte Carlo X-ray transport simulation of small-angle X-ray scattering instruments using measured sample cross sections. Choi, Mina; Ghammraoui, Bahaa; Badal, Andreu; Badano, Aldo. *Journal of Applied Crystallography*. Feb2016, Vol. 49 Issue 1, p188-194. 6p. DOI: 10.1107/S1600576715023924.

Volumetric Measurement of Residual Stress Using High Energy X-Ray Diffraction. Whitesel, R.; McKenna, A.; Wendt, S.; Graya, J. *AIP Conference Proceedings*. 2016, Vol. 1706 Issue 1, p1-8. 8p. 2 Diagrams, 1 Chart, 7 Graphs. DOI: 10.1063/1.4940584.

***In-situ* X-ray diffraction study on the structural reversibility of lithium nickel cobalt oxide in a broad electrochemical window of 1.35–4.3** V. Wang, Pengfei; Li, Peng; Yi, Ting-Feng; Yu, Haoxiang; Lin, Xiaoting; Qian, Shangshu; Zhu, Yan-Rong; Shui, Miao; Shu, Jie. *Electrochimica Acta*. Feb2016, Vol. 190, p248-257. 10p. DOI: 10.1016/j.electacta.2015.12.200.

Stimulated Compton scattering of soft X-ray radiation by hydrogen. Bachau, H.; Dondera, M.; Florescu, V. *Journal of Modern Optics*. Feb2016, Vol. 63 Issue 4, p402-410. 9p. DOI: 10.1080/09500340.2015.1064549.

Liquid contrabands classification based on energy dispersive X-ray diffraction and hybrid discriminant analysis. YangDai, Tianyi; Zhang, Li. *Nuclear Instruments & Methods in Physics Research Section A*. Feb2016, Vol. 808, p128-134. 7p. DOI: 10.1016/j.nima.2015.10.085.

Refinement of iron ore sinter phases: a silico-ferrite of calcium and aluminium (SFCA) and an Al-free SFC, and the effect on phase quantification by X-ray diffraction. Liles, David; Villiers, Johan; Kahlenberg, Volker. *Mineralogy & Petrology*. Feb2016, Vol. 110 Issue 1, p141-147. 7p. DOI: 10.1007/s00710-015-0411-5.

The promise of resonant inelastic X-ray scattering for modern Kondo physics. Hancock, J.N.; Jarrige, I. *Journal of Magnetism & Magnetic Materials*. Feb2016, Vol. 400, p41-46. 6p. DOI: 10.1016/j.jmmm.2015.08.017.

Deformation behavior of oriented β-crystals in injection-molded isotactic polypropylene by *in situ* X-ray scattering. Chen, Yanhui; Yang, Song; Yang, Haoqing; Zhong, Ganji; Fang, Dufei; Hsiao, Benjamin S.; Li, Zhongming. *Polymer*. Feb2016, Vol. 84, p254-266. 13p. DOI: 10.1016/j.polymer.2016.01.004.

Analysis of small-angle X-ray scattering data in the presence of significant instrumental smearing. Bergenholtz, Johan; Ulama, Jeanette; Zackrisson Oskolkova, Malin. *Journal of Applied Crystallography*. Feb2016, Vol. 49 Issue 1, p47-54. 7p. DOI: 10.1107/S1600576715023444.

Portable X-ray fluorescence spectroscopy as a rapid screening technique for analysis of TiO_2 and ZnO in sunscreens. Bairi, Venu Gopal; Lim, Jin-Hee; Quevedo, Ivan R.; Mudalige, Thilak K.; Linder, Sean W. *Spectrochimica Acta Part B*. Feb2016, Vol. 116, p21-27. 7p. DOI: 10.1016/j.sab.2015.11.008.

Determination of rare earth elements in combustion ashes from selected Polish coal mines by wavelength dispersive X-ray fluorescence spectrometry. Smolinski, Adam; Stempin, Marek; Howaniec, Natalia. *Spectrochimica Acta Part B*. Feb2016, Vol. 116, p63-74. 12p. DOI: 10.1016/j.sab.2015.12.005.

Determination of trace elements in freshwater rotifers and ciliates by total reflection X-ray fluorescence spectrometry. Woelfl, S.; Óvári, M.; Nimptsch, J.; Neu, T.R.; Mages, M. *Spectrochimica Acta Part B*. Feb2016, Vol. 116, p28-33. 6p. DOI: 10.1016/j.sab.2015.12.001.

Chemical mapping of mine waste drill cores with laser-induced breakdown spectroscopy (LIBS) and energy dispersive X-ray fluorescence (EDXRF) for mineral resource exploration. Kuhn, Kerstin; Meima, Jeannet A.; Rammlmair, Dieter; Ohlendorf, Christian. *Journal for Geochemical Exploration*. Feb2016, Vol. 161, p72-84. 13p. DOI: 10.1016/j.gexplo.2015.11.005.

X-ray micro tomography of three-dimensional embroidered current collectors for lithium-ion batteries. Aguiló-Aguayo, Noemí; Amann, Peter; Espiñeira, Pablo Pena; Petrasch, Jörg; Bechtold, Thomas. *Journal of Power Sources*. Feb2016, Vol. 306, p826-831. 6p. DOI: 10.1016/j.jpowsour.2015.10.039.

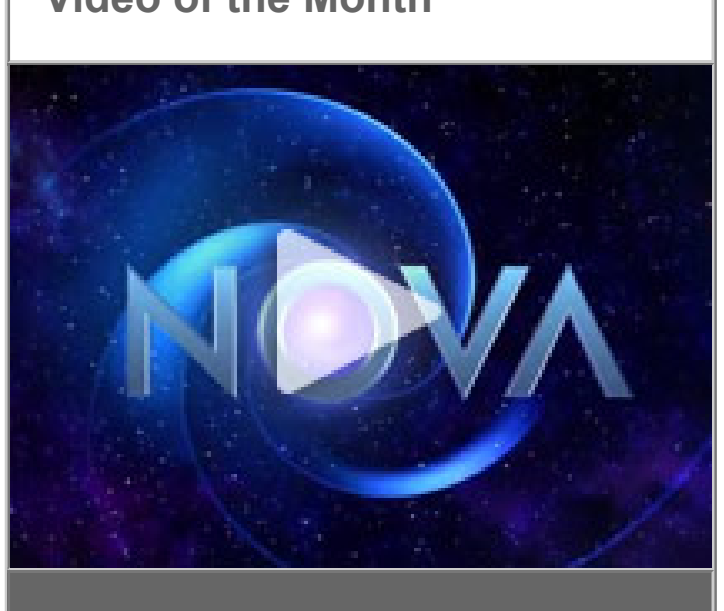
A carbon nanotube field-emission X-ray tube with a stationary anode target. Sharma, Anjli; Kim, Ho Seob; Kim, Dae-Wook; Ahn, Seungjoon. *Microelectronic Engineering*. Feb2016, Vol. 152, p35-40. 6p. DOI: 10.1016/j.mee.2015.12.021.

An iterative approach for compound detection in an unknown pharmaceutical drug product: Application on Raman microscopy. Boiret, Mathieu; Gorretta, Nathalie; Ginot, Yves-Michel; Roger, Jean-Michel. *Journal of Pharmaceutical & Biomedical Analysis*. Feb2016, Vol. 120, p342-351. 10p. DOI: 10.1016/j.jpba.2015.12.038.

Comparison of Raman and IR spectroscopy for quantitative analysis of gasoline/ethanol blends. Corsetti, Stella; McGloin, David; Kiefer, Johannes. *Fuel*. Feb2016, Vol. 166, p488-494. 7p. DOI: 10.1016/j.fuel.2015.11.018.

Improvement in X-ray stress measurement using Debye-Scherrer rings by in-plane averaging. Miyazaki, Toshiyuki; Fujimoto, Yohei; Sasaki, Toshihiko. *Journal of Applied Crystallography*. Feb2016, Vol. 49 Issue 1, p241-249. 8p. DOI: 10.1107/S160057671600128X.

Video of the Month



PBS NOVA: Inside Einstein's Mind

On November 25th, 1915, Einstein published his greatest work: general relativity. The theory transformed our understanding of nature's laws and the entire history of the cosmos, reaching back to the origin of time itself. Now, in celebration of the 100th anniversary of Einstein's achievement, NOVA tells the inside story of Einstein's masterpiece.

Now in its 43rd season, *Nova* is an American popular science television series produced by WGBH Boston. It is broadcast on Public Broadcasting Service (PBS) in the U.S., and in more than 100 other countries. The series has won many major television awards. Nova was created on March 3, 1974 by Michael Ambrosino, inspired by the BBC 2 television series *Horizon*. **Watch the video >**

Conferences and Workshops

Join Rigaku at future meetings

Rigaku will be sponsoring, attending or exhibiting at the following conferences and trade shows:

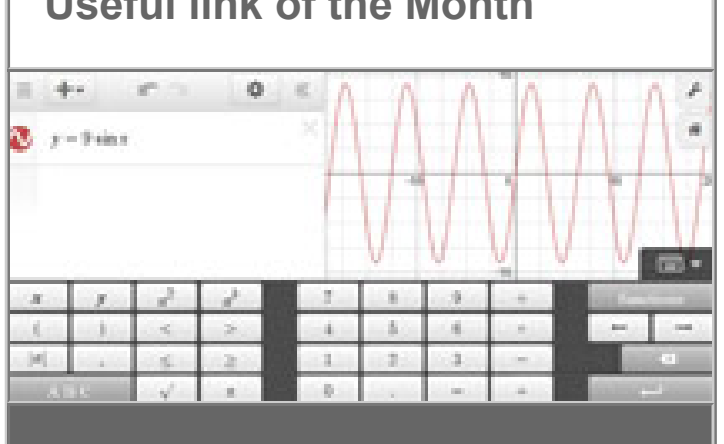
PITTCON
Atlanta, GA, USA
March 6 – 10, 2016

ACS – Spring
San Diego, CA, USA
March 13 – 17, 2016

APS American Physical Society
Baltimore, MD, USA
March 14 – 18, 2016

See the complete list >

Useful link of the Month



Desmos

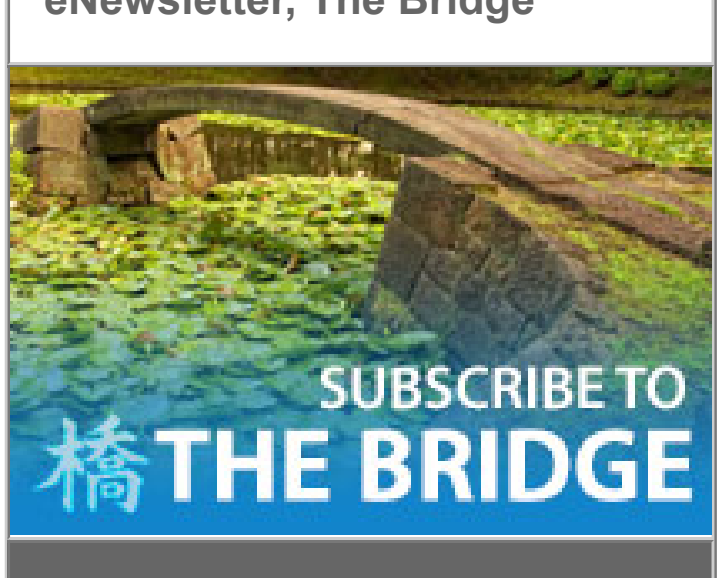
Desmos wants to help everyone learn math and love learning math. They created digital math tools and let the Internet take them to anyone who wants them. The best-in-class HTML5 Desmos graphing calculator, which millions around the world use for free, is worth a look. Sign in with your gMail account to save your work.
For more >

Planning to Submit a Grant?

Rigaku is happy to assist

If you are planning on submitting an instrument grant proposal, Rigaku will be happy to assist you. We can help you determine the correct instrument and configura-tion best suited for your analytical needs. **Start the process >**

Rigaku's Materials Analysis eNewsletter, The Bridge



Join us

Each month, Rigaku distributes two eNewsletters: *The Bridge*, which focuses on Materials Analysis, and *Crystallography Times*, which concentrates on life sciences. **Join us >**