

Elements

An International Magazine of Mineralogy, Geochemistry, and Petrology

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Iron in Earth Surface Systems

KEVIN G. TAYLOR and KURT O. KONHAUSER, Guest Editors

**A Major Player in Chemical
and Biological Processes**

Iron in Microbial Metabolisms

**Geomicrobiology of Iron
in Extreme Environments**

**Transport from the Continents
to the Open Ocean**

Ferruginous Oceanic Conditions

Iron in Marine Sediments

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Elements

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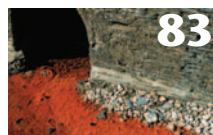
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Iron in Earth Surface Systems

Guest Editors: Kevin G. Taylor and Kurt O. Konhauser



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Iron in Earth Surface Systems: A Major Player in Chemical and Biological Processes

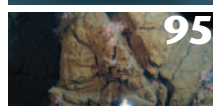
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Advances in our understanding of the origin and distribution in space and time of banded iron formations have increased our appreciation of the geological evolution of the Earth. Iron biogeochemistry has provided insights into not only past oceanic and sediment compositions but also current biogeochemical processes operating at or near the Earth's surface. The cover shows a polished slab of banded iron formation from the Hamersley Range, Western Australia. It is composed of alternating, millimeter-scale band of hematite (gray), chert (red), and tiger's eye (yellow). Field of view = 10 cm.

PHOTO COURTESY OF BARB DUTROW

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The Mineralogical Society of America is composed of individuals interested in mineralogy, crystallography, petrology, and geochemistry. Founded in 1919, the Society promotes, through education and research, the understanding and application of mineralogy by industry, universities, government, and the public. Membership benefits include special subscription rates for *American Mineralogist* as well as other journals, a 25% discount on Reviews in Mineralogy & Geochemistry series and Monographs, *Elements*, reduced registration fees for MSA meetings and short courses, and participation in a society that supports the many facets of mineralogy.

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The Mineralogical Society of Great Britain and Ireland is an international society for all those working in the mineral sciences. The Society aims to advance the knowledge of the science of mineralogy and its application to other subjects, including crystallography, geochemistry, petrology, environmental science and economic geology. The Society furthers its aims through scientific meetings and the publication of scientific journals, books and monographs. The Society publishes *Mineralogical Magazine* and *Clay Minerals*. Students receive the first year of membership free of charge. All members receive *Elements*.

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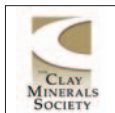
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The Mineralogical Association of Canada was incorporated in 1955 to promote and advance the knowledge of mineralogy and the related disciplines of crystallography, petrology, geochemistry, and economic geology. Any person engaged or interested in these fields may become a member of the Association. Membership benefits include a subscription to *Elements*, reduced cost for subscribing to *The Canadian Mineralogist*, a 20% discount on short course volumes and special publications, and a discount on the registration fee for annual meetings.

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The Clay Minerals Society (CMS) began as the Clay Minerals Committee of the US National Academy of Sciences – National Research Council in 1952. In 1962, the CMS was incorporated with the primary purpose of stimulating research and disseminating information relating to all aspects of clay science and technology. The CMS holds an annual meeting, workshop, and field trips, and publishes *Clays and Clay Minerals* and the CMS Workshop Lectures series. Membership benefits include reduced registration fees to the annual meeting, discounts on the CMS Workshop Lectures, and *Elements*.

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The Geochemical Society (GS) is an international organization founded in 1955 for students and scientists involved in the practice, study and teaching of geochemistry. Our programs include co-hosting the annual Goldschmidt Conference™, editorial oversight of *Geochimica et Cosmochimica Acta (GCA)*, supporting geochemical symposia through our Meeting Assistance Program, and supporting student development through our Student Travel Grant Program. GS annually recognizes excellence in geochemistry through its medals, lectures and awards. Members receive a subscription to *Elements*, special member rates for *GCA* and *G-cubed*, and publication and conference discounts.

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The European Association of Geochemistry was founded in 1985 to promote geochemical research and study in Europe. It is now recognized as the premiere geochemical organization in Europe encouraging interaction between geochemists and researchers in associated fields, and promoting research and teaching in the public and private sectors.

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The International Association of Geochemistry (IAGC) has been a pre-eminent international geochemical organization for over 40 years. Its principal objectives are to foster cooperation in the advancement of applied geochemistry by sponsoring specialist scientific symposia and the activities organized by its working groups and by supporting its journal, *Applied Geochemistry*. The administration and activities of IAGC are conducted by its Council, comprising an Executive and ten ordinary members. Day-to-day administration is performed through the IAGC business office.

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The Société Française de Minéralogie et de Cristallographie, the French Mineralogy and Crystallography Society, was founded on March 21, 1878. The purpose of the Society is to promote mineralogy and crystallography. Membership benefits include the "bulletin de liaison" (in French), the *European Journal of Mineralogy*, *Elements*, and reduced registration fees for SFMC meetings.

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The Association of Applied Geochemists is an international organization founded in 1970 that specializes in the field of applied geochemistry. It aims to advance the science of geochemistry as it relates to exploration and the environment, further the common interests of exploration geochemists, facilitate the acquisition and distribution of scientific knowledge, promote the exchange of information, and encourage research and development. AAG membership includes the AAG journal, *Geochemistry: Exploration, Environment, Analysis*; the AAG newsletter, *EXPLORE*; and *Elements*.

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The Deutsche Mineralogische Gesellschaft (German Mineralogical Society) was founded in 1908 to "promote mineralogy and all its subdisciplines in teaching and research as well as the personal relationships among all members." Its great tradition is reflected in the list of honorary fellows, who include M. v. Laue, G. v. Tschermak, P. Eskola, C.W. Correns, P. Ramdohr, and H. Strunz. Today, the Society especially tries to support young researchers, e.g. to attend conferences and short courses. Membership benefits include the *European Journal of Mineralogy*, the DMG Forum, *GMit*, and *Elements*.

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The Società Italiana di Mineralogia e Petrologia (Italian Society of Mineralogy and Petrology), established in 1940, is the national body representing all researchers dealing with mineralogy, petrology, and related disciplines. Membership benefits include receiving the *European Journal of Mineralogy*, *Plinius*, and *Elements*, and a reduced registration fee for the annual meeting.

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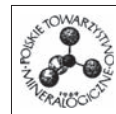
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The International Association of Geoanalysts is a worldwide organization supporting the professional interests of those involved in the analysis of geological and environmental materials. Activities include the management of proficiency testing programmes for bulk rock and micro-analytical methods, the production and certification of reference materials and the publication of the Association's journal, *Geostandards and Geoanalytical Research*.

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The Polskie Towarzystwo Mineralogiczne (Mineralogical Society of Poland), founded in 1969, draws together professionals and amateurs interested in mineralogy, crystallography, petrology, geochemistry, and economic geology. The Society promotes links between mineralogical science and education and technology through annual conferences, field trips, invited lectures, and publishing. Membership benefits include subscriptions to *Mineralogia* and *Elements*.

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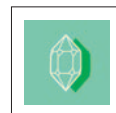
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The Sociedad Española de Mineralogía (Spanish Mineralogical Society) was founded in 1975 to promote research in mineralogy, petrology, and geochemistry. The Society organizes annual conferences and furthers the training of young researchers via seminars and special publications. The *SEM Bulletin* published scientific papers from 1978 to 2003, the year the Society joined the *European Journal of Mineralogy* and launched *Macla*, a new journal containing scientific news, abstracts, and reviews. Membership benefits include receiving the *European Journal of Mineralogy*, *Macla*, and *Elements*.

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The Swiss Society of Mineralogy and Petrology was founded in 1924 by professionals from academia and industry and by amateurs to promote knowledge in the fields of mineralogy, petrology and geochemistry and to disseminate it to the scientific and public communities. The Society coorganizes the annual Swiss Geoscience Meeting and publishes the *Swiss Journal of Geosciences* jointly with the national geological and paleontological societies.

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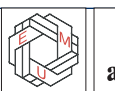
The Meteoritical Society is an international organization founded in 1933 for scientists, collectors, and educators to advance the study of meteorites and other extraterrestrial materials and their parent asteroids, comets, and planets. Members receive our journal, *Meteoritics and Planetary Science*, reduced rates for *Geochimica et Cosmochimica Acta*, which we cosponsor, the *Meteoritical Bulletin*, and *Elements*. We organize annual meetings, workshops, and field trips, and support young planetary scientists worldwide. Through our medals and awards, we recognize excellence in meteoritics and allied fields.

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The International Mineralogical Association, the European Mineralogical Union, and the International Association for the Study of Clays are affiliated societies of *Elements*. The affiliated status is reserved for those organizations that serve as an "umbrella" for other groups in the fields of mineralogy, geochemistry, and petrology, but that do not themselves have a membership base.



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THE MASTERY OF IRON



David J. Vaughan

Kipling's poem is a powerful reminder that iron, the subject of this issue of *Elements*, is a raw material as essential to industry today as it was three hundred years ago. In 1709, Abraham Darby initiated the industrial revolution by being the first to use coke in iron smelting, which he accomplished at his ironworks on the banks of the River Severn in Shropshire, England. Iron accounts for over 90 percent of all metals consumed annually and, over recent decades, there has been a dramatic increase in the total amount of iron annually extracted from the Earth. In just the fifteen years between 1994 and 2009, global iron production grew from 1000 million to over 2200 million metric tons. Iron is one of the six most abundant metals in the Earth's crust, so there is no danger of running out of minable deposits of iron. However, most of the iron produced for modern industry is only useful when alloyed with one or more of a number of other metals that act to increase its strength, machinability or corrosion resistance, or provide it with special properties, such as those associated with magnetism.

Whereas iron is still, as Kipling implies, the 'backbone of industry', it is ironic that geochemically scarce metals, such as chromium, titanium, cobalt, nickel, and tungsten, are essential for using that iron. Furthermore, in addition to metals for alloying with iron, a growing number of scarce and rare metals are essential raw materials for modern technologies. Examples include lithium for batteries, germanium and indium for computers, and tantalum for mobile phones. It is becoming apparent that shortages may soon arise in the supplies of certain of these metals; indeed, they are acquiring the kind of strategic importance previously only associated with reserves of oil and gas. The rare earth elements (REE) are a good example: they are used in small amounts in everything from high-powered magnets to batteries and electronic circuits. The need for these metals is rapidly growing because of their roles in clean-energy technologies (lanthanum for hybrid car batteries, neodymium for permanent magnets in wind turbines, and europium for

energy-efficient lighting). At present, China controls 97% of global REE production. In October 2010, China cut its exports of REE by more than 70% over the previous year, disrupting manufacturing in the USA, Europe and Japan and forcing price rises of 40% or more. According to experts, these cuts were related more to the needs of the Chinese to satisfy their own growing internal market than to an attempt to profit from their near monopoly. Such developments raise the spectre of future international economic or even military conflicts arising over the supplies of rare metals.

The role of geoscientists in addressing the issues concerned with the supplies of strategic metals will be obvious to the readers of *Elements* magazine. Our community has been involved since the beginning in the application of science to mineral exploration, mining, mineral processing and metallurgy. These contributions are more important than ever, and are now complemented by the

*Gold is for the mistress
Silver for the maid
Copper for the craftsman, cunning
at his trade*

*'Good' said the Baron,
sitting in his hall,
But iron, cold iron, is
master of them all*

—Rudyard Kipling
(1835–1936)

efforts we make towards the 'responsible' exploitation and utilization of Earth resources. In the cycle of mining, ore processing, metal utilization, and either recycling or safe disposal of the end products (and especially also of the wastes associated with mining), geoscientists need to play a central role in ensuring that we do not cause irrevocable damage to our environment. With a world population now approaching seven billion people, and predicted to grow to around nine billion by 2050, demand for metals is bound to further increase. Any return to 'simpler' ways of living that would not require such resources is unrealistic. Without mechanized food production and distribution, for example, there would be mass starvation around the globe.

This is my last editorial as a principal editor of *Elements* magazine, and thinking back over my previous editorials I recognize one central theme. It is that the community of Earth scientists, particularly those whose interests are focused on mineralogy–petrology–geochemistry, should be 'centre stage' in addressing many of the most pressing concerns that threaten the health and even the survival of future generations. These problems include the safe containment or clean-up of hazardous wastes (especially nuclear waste); pollution of the atmosphere impacting on human health; climate change, whether of human or other origins; and, in the context of the present article, secure supplies of many raw materials, including 'rare' metals. In the debates about how best to tackle these concerns, we need to be sure that our arguments are based on rigorous and objective science and, above all, that our voices are heard.

David J. Vaughan

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THIS ISSUE

Iron is one of the most abundant elements in the Earth's crust, an element essential to many forms of life, and a material civilizations have used for thousands of years to fashion tools and arms. In assembling this issue, Guest Editors Kevin Taylor and Kurt Konhauser have focused on the role of iron in Earth surface environments, past and present, and particularly on how iron minerals in sediments can be used as proxies for the composition of ancient oceans. Indeed, for an abundant element that at first glance seems mundane, iron has enormous potential as a tracer of Earth evolution.

BREAKING NEWS

On March 11, a horrified world watched the images of devastation following the earthquake and tsunami that struck the northeast coast of Japan. Since then, we have all admired the resilience and courage of the Japanese people amid the destruction. We continue to watch daily the events unfolding at the Fukushima nuclear power station in the aftermath of the tsunami.

As the focus in Japan moved towards the multi-tiered crises at the nuclear power reactors, we recognized that there was a dearth of accessible information available particularly concerning the spent fuel in the cooling ponds. *Elements* has helped to fill this void. The Nuclear Fuel Cycle issue we published in 2006 (volume 2, number 6) was made freely available through our website (www.elementsmagazine.org) and on GeoScienceWorld (www.elements.geoscienceworld.org). Announcements were made to our members, who forwarded the information far and wide: to press and media outlets, to our sister organizations, and to policy makers.

As a follow-up to the events in Japan, our inaugural "Breaking News" article, by Rod Ewing, who was guest editor of the Nuclear Fuel Cycle issue, covers the situation at Fukushima as of late March (see pages 77 and 78 of this issue).

To our Japanese colleagues, we at *Elements* extend our heartfelt thoughts in this time of crisis.

THANKS DAVID

David Vaughan has been a big supporter of *Elements*, starting from the moment he heard about the magazine concept from Rod Ewing. In the very early days of *Elements*, while David was on the Advisory Board, he volunteered to organize an issue on arsenic, for which he was guest editor (volume 2, number 2). The Arsenic issue is our second most heavily cited number and is a popular teaching tool in environmental science. His introductory article for that issue is a perfect example of what we hope all lead articles for *Elements* will be like. When invited to join the editorial team, he did not hesitate. He has now completed his term as principal editor, and we thank him for his enthusiasm and judgment on behalf of *Elements*. During his term, he was in charge of the following issues: Nanogeoscience, Gems, Mineral Magnetism, Mineral Evolution, Atmospheric Particles, and Iron in Earth Surface Systems. In his editorials, he has encouraged the Earth science community to be center stage in the ongoing debates in several issues the world is currently facing.

Pierrette Tremblay
Managing Editor

LIFE IN UREY'S LAB

Congratulations on one of the most informative, interesting, and germane issues of *Elements* (Cosmochemistry; volume 7, number 1): it invoked a lot of memories. In the early 1960s I worked for Harold Urey in La Jolla at Scripps Institute right after he moved to California from Chicago. I was a lab assistant (actually a lab rat) for several years between 1959 and 1969 as I worked my way through high school and then college, first as a chemist and later as a programmer. Urey's work on meteorites and terrestrial rocks made his lab seem like the sphere of a Van de Graaff generator. I went to most of the seminars he and his colleagues presented and to several parties given for and by him; it was the most exciting time of my life. I experienced many ways to do science and learned a great deal. Urey was involved in designing and building from scratch a mass spectrometer (that machine ended up at the U. of Minnesota), which was used for several years. This instrument and the neutron activation analysis technique were used to measure the ratios of various isotopes. It was the NAA that got me interested in learning about computers since I did most of the calculations manually on a "Monroe-matic" (a beast with more moving parts than you could shake a stick at), and it took forever. Scripps had taken delivery of a CDC-1604, and when I walked into the computer room (security was of no concern back then), with all its blue glass and aluminum, it was love at first sight. I asked the operator how I could use this amazing machine, and he gave me three manuals, pointed to a key punch, explained how to load my cards into the reader, and said, "Good luck"; I had to teach myself FORTRAN. That was in 1964. I just retired last month from my career as a computer programmer, but I have kept my interest in all the other fields that were useful back in the days with Urey. As a member of the Geochemical Society (which is how I get *Elements*), your magazine has helped me inexpensively keep abreast of new ideas on the origin of the Solar System. Thanks for a great publication!

Richard A. J. Cox
Saratoga, CA, USA

OXIDE MINERALS AND THE COSMOS

May I thank you and your authors for the excellent edition of *Elements* on Cosmochemistry. I have been reviewing the main oxide minerals (for Deer, Howie and Zussman, volume 5a, to be published shortly) and I found the developments in extraterrestrial mineralogy to be among the most interesting new facets of oxide mineralogy. Periclase, corundum, rutile, perovskite, hematite, ilmenite, armalcolite (of course), spinel, magnetite, maghemite, ulvöspinel and chromite have all been recorded from beyond the Earth, and isotopic studies of these minerals have provided evidence about presolar history, the age of the Solar System, and the development of the planets and asteroids. This is a remarkable achievement, the ultimate in "distance learning." I have read your articles from beginning to end with great interest and look forward to reading them again. It is important to inform mineralogists of the way in which mineralogy is applied in other disciplines. Your issue of *Elements* provides a concise description of the place of mineralogy in cosmochemistry and will serve as an introductory text for students and researchers alike.

Dr John F. W. Bowles
The University of Manchester, UK

BEYOND TRIAGE AT THE FUKUSHIMA
DAI-ICHI NUCLEAR POWER STATION

Rod Ewing

On Friday afternoon, March 11, Fukushima prefecture in Japan was shaken by a magnitude 9.0 earthquake whose hypocenter was 24 km deep and about 160 km to the northeast, offshore Honshu Island. At the Fukushima Dai-ichi nuclear power station, the earthquake caused three of the six nuclear reactors to “SCRAM” (Safety Control Rod

Axe Man: a process of inserting control rods to absorb neutrons and shut down the nuclear chain reaction). Three of the reactors were already in shutdown mode for inspection and maintenance. The design basis for the six nuclear power plants was for an earthquake of magnitude 8.2. The earthquake caused the operating plants to enter a standard shutdown cooling mode in order to remove the heat generated by the radioactive decay of fission product elements. This residual heat is only 3 to 6 percent of the heat generated while the fuel is in the reactor, but the rate of energy release is still on the scale of several million watts per ton of fuel. This heat must be removed in order to prevent the nuclear fuel, mainly UO_2 , and supporting structural elements from melting in the core of the reactor. The melting point of UO_2 is approximately 2900 °C.

Within an hour, however, a tsunami, variously reported as between 8 and 14 meters high, hit the six nuclear power plants at Fukushima Dai-ichi. The design basis for a tsunami hitting the plants was reported to be 5.7 meters. The tsunami cut off electricity from the backup diesel generators that powered the water pumps used to maintain water circulation around the cores of the reactors; the failure of the generators was probably due to a loss of fuel. Backup battery power was used to operate the pumps, but this source lasted only about eight hours and then went dead, creating a complete station blackout. During the next ten days, the world followed the heroic efforts of plant operators, fire fighters, and the Self-Defense Forces to deal with an escalating series of potentially catastrophic events resulting from power loss: melting cores in three of the reactors; probably three hydrogen explosions; and the loss of water from the pools in which spent or used fuel is stored, which could lead to rising temperatures and ultimately ignition of the zirconium metal alloy that clads the fuel elements (this is an exothermic reaction and is written as $\text{Zr} + 2\text{H}_2\text{O} \rightarrow \text{ZrO}_2 + 2\text{H}_2$). In an extraordinary effort to cool both the reactor cores and the used fuel in the pools, seawater was pumped into the reactor cores and the used-fuel storage pools. Fluctuating levels of radiation, as well as smoke and fires, hampered the effort to stabilize the conditions of the reactors and storage pools. The Japanese government evacuated hundreds of thousands of people within a 20 km radius. All of these events took place against a backdrop of devastation and death that will probably claim the lives of several tens of thousands of Japanese.

As I write today, the situation remains serious. The sequence of events at the Fukushima Dai-ichi power plant, up to March 30, are beautifully illustrated on the accompanying figure provided by Rama Hoetziein. The situation for the nuclear power plants and fuel storage pools has been stabilized by the initial triage, but it will take many weeks of intensive care before the release of radioactivity to the environment is stopped. After this period, the authorities will have to reconstruct the sequence of events, estimate the damage, evaluate the environmental impact, and develop strategies for remediation. This last step does not have the nerve-jangling attraction of a possible meltdown or explosion at a nuclear power plant, but over the long term the environmental impact of the destroyed nuclear reactors may be just as serious. Each

reactor core contains approximately 100 metric tons (tonnes) of fuel (mostly UO_2). The fuel from reactor #4 had recently been moved to a spent-fuel pool. Thus, there are approximately 500 tonnes of nuclear fuel in the reactors (three of which have at least a partially melted core due to the loss of coolant and resulting high temperatures). Each reactor has its own spent- or used-fuel storage pool, with estimates ranging from 50 to 150 tonnes of used fuel per pool; thus, there are an estimated 600 tonnes of used fuel in these storage pools. A separate shared pool contains approximately 1000 tonnes of fuel. Importantly, most of the nuclear fuel is located in storage pools, not in the reactors. In addition, 70 tonnes of nuclear materials are in dry storage on site. In total, there are some 1700 tonnes of nuclear fuel at Fukushima Dai-ichi—a site of “inadvertent, near-surface geologic disposal.”

The state of the fuel is not known, but with the mechanical and thermal shocks to the spent-fuel pools, one expects that much of the metal cladding (less than a millimeter thick) has failed. For average burnups of 40–50 megawatt-days/kilogram of uranium, a measure of the amount of energy extracted from the uranium fuel, one can roughly estimate that about 3% of the uranium has been fissioned and 1% has been converted to transuranium elements—mostly plutonium, with lesser amounts of neptunium, americium, and curium—formed by neutron capture and decay reactions. Much has been said about the mixed oxide fuel (MOX) in reactor #3, but only about 5% of the fuel assemblies in the reactor were MOX fuel elements. The MOX fuel is reported to have contained ~6% plutonium as a mixture of plutonium and uranium oxide. In total, the site has approximately 15 tonnes of plutonium that is in the nuclear fuel, mostly ^{239}Pu with a half-life of 24,100 years. The very strong radiation field, mainly gamma and beta radiation, and the source of most of the thermal energy, comes from short-lived radionuclides, such as ^{137}Cs and ^{90}Sr , which have half-lives of about 30 years.

The most important environmental decision to be made at the site will be whether to try to remove all the fuel for safe storage and disposal, such as was done with the fuel after the melting of the core at Three Mile Island in 1979, or whether to simply seal the reactors in a concrete sarcophagus, as was done at Chernobyl. It may well be that much of the fuel can be removed, particularly from the least affected storage pools, but some may finally remain on site. This decision will have to be guided by a thorough understanding of the “mineralogy” of the fuel and the geochemistry of the fission product elements, particularly ^{135}Cs and ^{129}I , both of which have very long half-lives, and the actinides, mainly plutonium, in this near-surface environment.

Natural events—an earthquake and a tsunami—caused this devastating nuclear accident, and natural geochemical and hydrological processes will determine its environmental impact.

The reader should note that much of this information has been pieced together from a variety of sources, including newspaper reports. The numbers I quote are only rough estimates and are given so that readers can appreciate the scale of the accident. An exact understanding of what happened at the Fukushima Dai-ichi nuclear power station will be developed over the next year.

Rodney C. Ewing

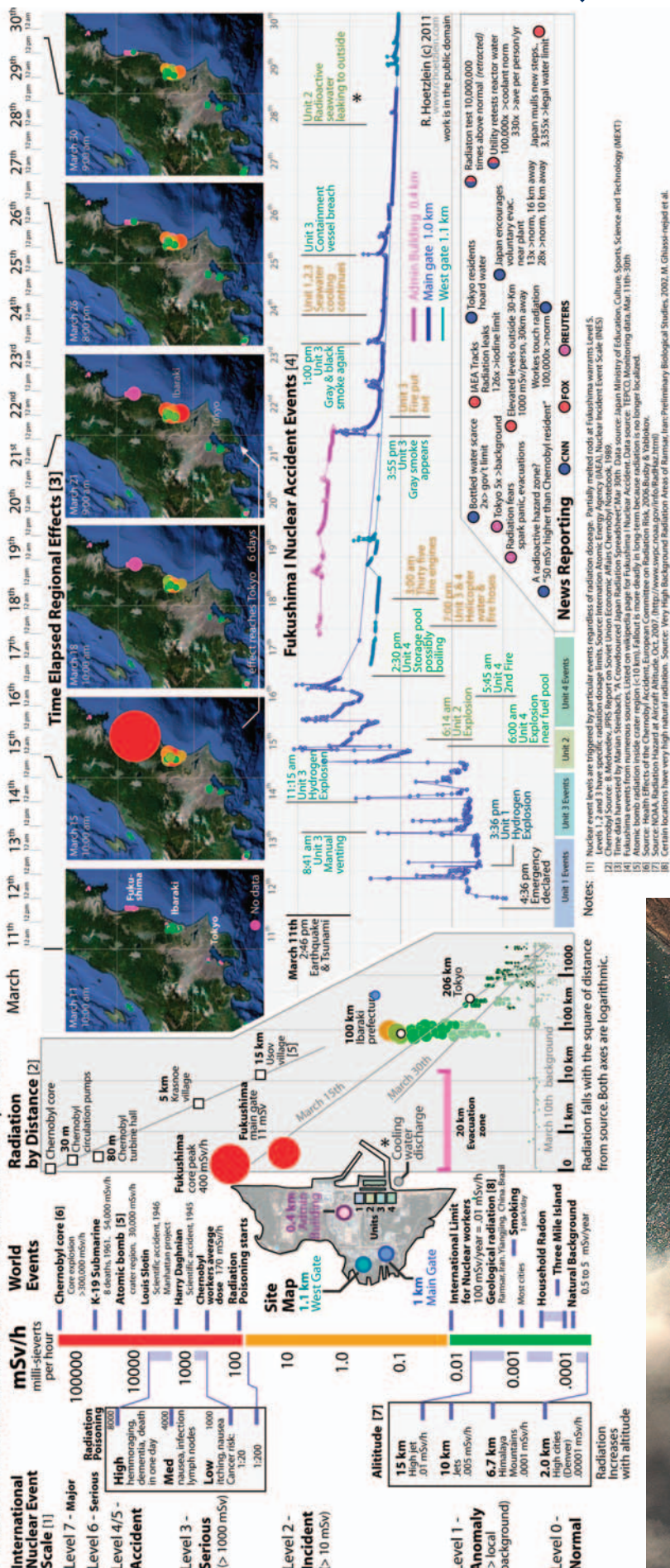
Visiting Professor at the Center for International Security and Cooperation, Stanford University

Suggested Readings

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Fukushima Nuclear Accident - Radiation Comparison



This visualization of the relation between radiation doses reported at the Fukushima Dai-ichi nuclear power station and typical exposures to radiation was created by Rama Hoetzelin. The graphical representation also includes a time series visualization of regional effects, a local site map, and data from selected news reports throughout March 30. All units are converted to mSv/h (millisieverts per hour) and plotted on a logarithmic scale. Normal background, chest CT scans, and food levels are usually expressed in mSv/y (millisieverts per year). Higher radiation doses, such as those found in nuclear disasters, are commonly given in Sv/h (sieverts per hour).

From an analysis of data in 64 media reports, Hoetzelin concludes that news reports have presented an inconsistent view of the doses and events. The most significant problem appears to be in the correct reporting of radiation levels. Reported radiation doses have been inconsistent in terms of measurement, the radiation levels used for comparison, and radiation background levels.

We extend our sincere thanks to Rama C. Hoetzlein, who made the graphic available for reprint as part of this Breaking News article. Created March 30, 2011, an extensive analysis of this visualization is available at www.rchoetzlein.com/theory/. PUBLIC DOMAIN DOCUMENT

© Satellite image of the Fukushima nuclear power station taken on March 14. Several images taken at different dates are available at www.digitalglobe.com/index.php/27/Sample+Imagery+Gallery. © DIGITALGLOBE



SEEING SINGLE ATOMS – THE LAST FRONTIER

The Elements
Toolkit

A thorough knowledge of a given natural process often requires a detailed understanding of a suite of mechanisms operating across length scales spanning many orders of magnitude. An example of this phenomenon involves the single seismic event: (1) stress failure is triggered at the micro-scale, (2) mechanical rupture occurs along a fault plane at the kilometre scale, and (3) energy is dissipated at the global scale. For crystallographers and mineralogists, this “scaling factor” challenge manifests itself most acutely at the shortest length scale. Key grain boundary and mineral reaction mechanisms operate at atomic to nanometre scales, and this fact drives the need for analytical techniques with ever improving spatial resolutions. Compared to a few decades ago, the array of analytical technologies now available is impressive: μ -XRF (spatial resolution around 100 μm), laser ablation ICP-MS (100 to 10 μm), SIMS (20 μm to 100 nm), electron probe and SEM quantification (1 μm to 100 nm), Auger Microprobe (10 nm), and TEM (down to 1 nm or better). Each of these techniques has its advantages and drawbacks. Nonetheless, detailed characterisation of geo-materials is now technically possible at length scales spanning the nanometre to hand specimen range and beyond. The only technology lacking is the ability to evaluate the position and identity of single atoms.

Here I present the 3-dimensional atom probe, an emerging technology that may fill the one remaining gap in our analytical capabilities. Also known as the tomographic atom probe, this high-tech method involves the “evaporation” of individual atoms from the end of an extremely sharp tip made of the material being investigated. A sharp tip with a diameter on the order of <100 nanometres is essential as this provides the extremely high field gradients (on the order of 10^{10} V/m) needed for field emission to occur. Projecting the emitted atoms, whether from a desorbed imaging gas or from the sample itself, in combination with a 10^6 magnification between the sample and the imaging detector, allows atomic-scale resolution using a phosphorus screen and a simple hand lens.

Atom probe tomography (APT) had its roots six decades ago when Erwin Müller (1951), then at the Kaiser-Wilhelm-Institute in Berlin, described his theory of the field ion microscope. His device relied on the emission of adsorbed hydrogen molecules, which were subsequently desorbed from the sample tip; these protons were then accelerated onto a phosphorus screen, thereby producing a very weak optical signal that could be observed in the completely darkened laboratory. The device gave information only about the crystallographic structure of the material under investigation, and Müller estimated the spatial resolution of his method to be around 2 or 3 nm, which was certainly impressive for its time. That same year, Müller moved to take up a faculty position at Pennsylvania State College (now University) where he established a research group working on field ion and field electron microscopy.

The next breakthrough came in October 1955 when Kanwar Bahadur, a PhD student in Müller’s group, accomplished the first demonstration of lattice-scale resolution using a cooled tip in conjunction with helium imaging gas. Two similar experiments had previously been carried out by the group with limited success. It was thanks largely to the tenacity of Bahadur that success was achieved after he devised minor modifications to both the apparatus and the all-critical tip-preparation procedure (FIG. 1). These busy days in the Penn State lab are described in detail by Melmed (1996) and Kumar (2005). Müller went on to rapidly publish a number of single-author articles related to this achievement, while Bahadur obtained his doctorate in 1956 before returning to his former institute in New Delhi.

Until the mid-1960s the field ion microscope was limited to atomic-scale structural studies. Adsorbed ions from a low-pressure imaging gas were introduced into the sample chamber; these ions were subsequently



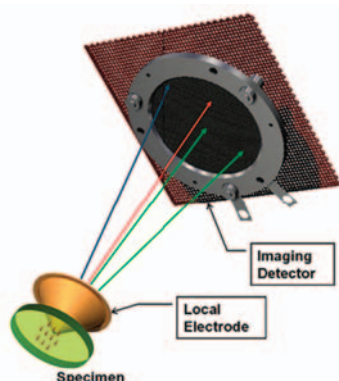
FIGURE 1 Historical photograph of Kanwar Bahadur at the controls of the original 1955 field ion microscope (from Kumar 2005, with permission from *Current Science*)

evaporated and ionized through the field emission mechanism. Frequently the sample atoms were not even involved in the actual imaging and, except in the case of pure substances, there was no way of knowing which elements were present. This changed when Müller et al. (1968) coupled a field ion microscope to a time-of-flight (ToF) mass spectrometer, creating the first true atom probe capable of identifying specific elements in the sample material. A key obstacle that had to be overcome was the requirement of ToF mass spectrometry to know with high certainty when a given ion was ejected from the sample, the event against which an ion’s flight time would be measured. This challenge was solved by pulsing the voltage applied to the sample tip, increasing the electric potential from some base-level voltage to above the threshold at which field emission can occur. In order to prevent the undesired continuous emission of ions, the voltage then had to be rapidly dropped back below the emission threshold, this entire sequence lasting perhaps 3 ns. By recording the sequence of atomic compositions originating from each single locale, it was possible to build a 3-dimensional map of the sample. Despite the limited field of view of <2 nm provided by this apparatus, it still can be seen as the first true APT instrument capable of determining both the structure and chemistry of a sample material, albeit limited to assessing only around 50,000 atoms (Miller and Forbes 2009).

Major limitations, however, still hampered the technology. The requirement that the voltage on the specimen tip be rapidly pulsed limited analyses to electrical conductors: semiconductors and insulators remained off limits. This situation changed in the 1970s (see review by Kelly and Miller 2007) with the realisation that short-duration laser pulses, focused on the sample tip, could be used to excite sample atoms to above the energy threshold at which field emission ionization occurs. Tsong (1978) reported photon-stimulated field ionization from a metal tip. Kellogg and Tsong (1980) were the first to successfully couple a ToF system to a pulsed-laser evaporation source. This new system had two major advantages over earlier pulsed-voltage field evaporation techniques. First, it brought the analysis of semiconductors into the realm of the possible. Second, it largely eliminated the broad energy distributions inherent to the emission of ions during either the voltage-pulse rise or drop that degrades the ToF spectrum. In hindsight, the major advance of photo-assisted field emission did not initially have many followers: lasers were expensive and difficult to align, and the thermal pulse was too protracted to give really precise start times for the emission process. In the early 2000s, more robust laser systems were developed. Also, the development of ultrafast lasers greatly improved the time resolution of the affiliated ToF spectrometer because the duration of thermally coupled emission could be reduced to below a few tens of picoseconds.

Another drawback of APT technology was the many problems associated with specimen preparation. Electropolishing had long been a preferred option, but it was not site specific and it generally required a

FIGURE 2 Basic layout of a local-electrode ion optical design. IMAGE COURTESY OF CAMECA



wire-like geometry for the starting material (see also Kelly and Miller 2007); also, the taper angle and diameter of the tip were difficult to control. In the 1980s, new ion-based methods of sample preparation were devised, and focused ion beam (FIB) technology was developed. The FIB technique has now become a must-have tool for many TEM applications, as the area to be investigated can be precisely selected and the geometry of the final specimen – whether a foil or a tip – can be controlled with nanometre precision. The first application of FIB sample preparation to APT technology was reported by Waugh et al. (1984), which opened up an entirely new world of possibilities for materials studies.

At the turn of the 21st century, the most common complaint of the atom probe user community was probably the slow data-collection rate: repetition rates were only on the order of 10 kHz, leading to the practical limit that a given experiment could only observe perhaps 10^6 ions. After correcting for ion-detection efficiency, this would translate into an available volume for a single analysis of only around 10^4 nm^3 . This tiny sample size excluded many applications for which APT was otherwise well suited. This began to change with the advent of “local electrode” technology whereby the specimen tip is positioned in front of a funnel-shaped extraction electrode with an aperture diameter of only a few tens of microns (Fig. 2). By placing the local electrode close to the sample – again we are speaking of a few tens of microns – much higher electric field gradients are achieved relative to “traditional” ion optic geometries operating at the same absolute potential. This yields a number of advantages when operating in pulsed-voltage mode, including better mass resolution for the ToF spectrometer and repetition rates well beyond 100 kHz (see Kelly et al. 1996). Other advantages of using a local electrode are that (1) the field of view of the system can exceed 100 nm, (2) it is possible to employ a planar microtip sample geometry and (3) one can load many sample tips on a single specimen holder as only the single tip in close proximity to the local electrode will undergo the field emission process. Thanks to this new ion optical design, experiments involving over 10^8 atoms and volumes in excess of 10^6 nm^3 became realistic (Fig. 3).

What challenges remain? Obviously it would be valuable to couple the local-electrode geometry with the pulsed-laser evaporation technique, thereby making state-of-the-art ion optics applicable to poorly conducting specimens. Exactly such studies have recently been reported.

FIGURE 3 An example of a 3-dimensional map of a semi-conductive structure acquired using pulsed laser APT. IMAGE COURTESY OF CAMECA

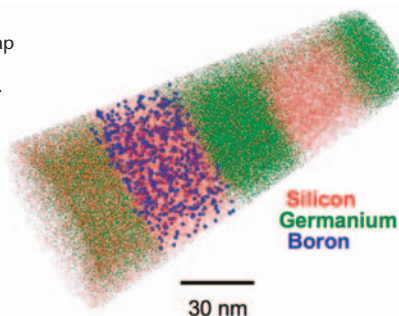


FIGURE 4 Cameca's laser-assisted atom probe, like the one used by Bachhav et al. (2011) to investigate wüstite. This particular instrument design has recently gone out of production, having been superseded by the “LEAP 4000X” model. IMAGE COURTESY OF CAMECA

Bachhav et al. (2011) used a 350 fs pulse-width laser operating at 100 kHz (Fig. 4) to analyse iron oxide grains glued to tungsten supports, which were then subjected to ion milling. Using this approach, these authors were able to quantify the Fe/O ratio in non-conducting wüstite at atomic-scale resolution. Equally interesting is a report by Gordon and Joester (2011), who used laser-assisted APT to investigate the nano-scale properties of organic fibres buried within the tooth of the marine mollusc *Chaetopleura apiculata*. Their data reveal a previously undocumented level of hierarchy within this organic scaffold.

APT sample preparation remains rather tedious, requiring expensive technology. For electrically non-conducting samples, many analytical parameters still need to be optimized. Nonetheless, atomic-scale mapping of non-conductors is nearly within our grasp. So how long will it be before this highest of high-tech methods will be taken up in the Earth sciences? I suspect that it is only a matter of time ... and money.

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Elemental & Isotopic Microanalysis in Geosciences: The CAMECA Expertise



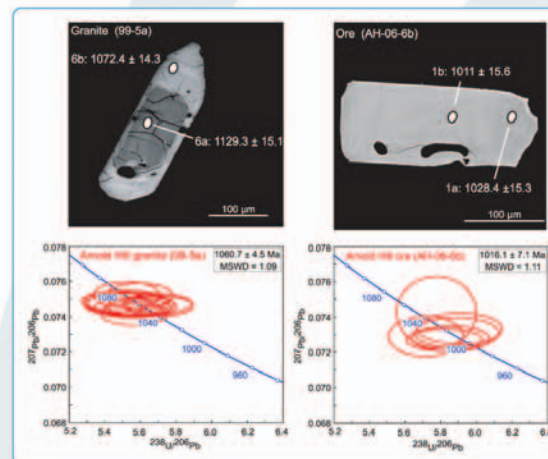
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Understanding the timing of Fe mineralization and hydrothermal alteration provides important information for ore petrogenesis, crustal fluids, and tectonic evolution of the host regions.

From P. Valley, J.M. Hanchar, M.J. Whitehouse, *GEOLOGY* 37 (2009).

U/Pb zircon geochronology: first direct dating of iron oxide-copper-gold (IOCG) mineralization. Hydrothermal zircon crystals were separated from four IOCG-type ore deposits associated with the Lyon Mountain granite (United States).



Top: Back-scattered electron images of zircon grains. Bottom: Concordia diagrams showing zircon dates. In situ U/Pb analyses of zircon grains from these low-Ti magnetite deposits (right figure, ore deposit) and their respective host granites (left figure, adjacent host rock) reveal that the ore mineralization post dates the emplacement of the granite by approximately 45 million years.



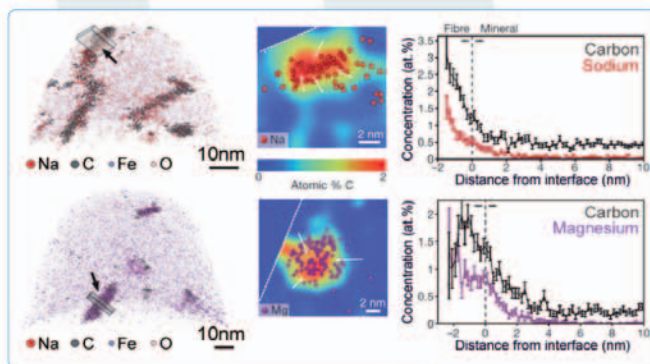
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Interfaces, nanograins, gradients, clusterings, segregations can be mass analyzed in the APT after extraction from bulk samples using standard Focused Ion Beam preparation technique.

Nanoscale chemical tomography of buried organic-inorganic interfaces in a chiton tooth: APT reveals three-dimensional chemical maps of organic fibres with a diameter of 5–10 nm in the surrounding nano-crystalline magnetite (Fe_3O_4) mineral in the tooth of a marine mollusc, the chiton *Chaetopleura apiculata*. Remarkably, most fibres co-localize with either sodium or magnesium.

From L.M. Gordon & D. Joester, *NATURE*, vol 469, 13 January 2011.



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Meet the Authors



Donald E. Canfield is a professor of ecology and director of the Nordic Center for Earth Evolution (NordCEE) in the Institute of Biology, University of Southern Denmark. Don has worked at understanding the modern cycles of iron and sulfur, the evolution of these cycles, and their interface with other element cycles, through geologic time. Don's work spans the range of microbial ecology, biogeochemistry, and geology. He may be found at any given time (when not sorting papers) in the lab, with his hands deep in mud, or banging on a rock. His favorite current research sites include meromictic Lake Cadagno in Switzerland and the oxygen-minimum zone off the coast of Chile.



Andreas Kappler is a professor of geomicrobiology at the University of Tübingen. He received an MSc/Diploma in chemistry and a PhD in microbiology from the University of Konstanz (Germany) and occupied postdoctoral positions at the ETH Zürich/EAWAG in environmental chemistry and at Caltech in geobiology. His research centers on the biogeochemical cycling of iron and humic substances and the consequences for the environmental fate of arsenic and organohalogens. He also studies the role of microbial iron oxidation in the deposition of Precambrian banded iron formations. He is on the editorial board of *Geobiology*, an associate editor of *PALAIOS*, and a councilor of the European Association of Geochemistry.



Kurt O. Konhauser is a professor of geomicrobiology in the Department of Earth and Atmospheric Sciences at the University of Alberta, Canada. He is editor-in-chief of the journal *Geobiology* and author of the textbook *Introduction to Geomicrobiology*, published by Wiley-Blackwell in 2007. His research is on the role of bacteria in modern mineral precipitation and elemental cycling and on how these same processes may have contributed to the preservation of early life forms and the formation of Precambrian banded iron formations. Other current interests include the use of Precambrian sediments as proxies for ancient seawater composition and evolution of the marine biosphere.



Joe H. S. Macquaker is an associate professor in the Department of Geology and Environmental Sciences at Memorial University, Newfoundland, Canada. Prior to moving to Canada in 2008, he had been on the faculty of the School of Earth, Environmental and Atmospheric Sciences at the University of Manchester, UK, where he had held various posts from reader to postdoctoral research associate over twenty years. In his research, he investigates how physical, chemical, and biological processes control lithofacies variability in organic carbon-rich, fine-grained sediments. He is currently examining how microbe-mediated respiratory processes interact with existing mineral phases to control diagenetic reactions in horizons associated with breaks in sediment accumulation.



Simon W. Poulton is a reader in biogeochemistry in the School of Civil Engineering and Geosciences at Newcastle University. Originally a geologist by training, his research interests now encompass the application of geochemical, biogeochemical, and isotopic techniques to address major issues related to the evolution of Earth's surface environment. He has paid particular attention to the operation of the global iron cycle and the development and application of iron speciation as a paleoredox tool. He has persistently returned to the study of anoxia in modern and ancient environments, not forgetting many enjoyable hours spent attempting to replicate such systems in the laboratory.



Robert Raiswell is an emeritus professor in the School of Earth and Environment, University of Leeds, where he has been since 1983. He received his PhD from Liverpool University in 1973 and has spent time as a visiting scientist at Yale, Georgia Institute of Technology, and the University of California, Riverside. His research is centered on the cycling of iron and sulfur in Earth surface environments, past and present. His current interests include the formation and bioavailability of iron nanoparticulates in glacial environments and the use of iron minerals to deduce the depositional environments of Proterozoic sediments. He was elected a fellow of the Geochemical Society in 2010.



Eric E. Roden is a professor of geoscience at the University of Wisconsin–Madison. He received his PhD from the University of Maryland and held postdoctoral positions at the U.S. Geological Survey and the Pacific Northwest National Laboratory. He joined the Department of Geoscience in Madison after twelve years in the Department of Biological Sciences at the University of Alabama–Tuscaloosa. His research is on aquatic biogeochemistry, specifically anaerobic microbial processes and the redox cycling of elements at the Earth's surface. He is a specialist in the ecology and physiology of iron-reducing and iron-oxidizing bacteria and has recently become involved in experimental studies of redox-driven fractionation of iron isotopes.



Bruce M. Simonson has been teaching and doing research with undergraduates at the Oberlin College Geology Department since 1979. He received his BA from Wesleyan University in 1972, then mapped in Central America for 2 years. He obtained his PhD from Johns Hopkins University where he was trained as a sedimentologist and acquired an enduring interest in surface environments on the early Earth. This led to field and petrographic studies of clastics, carbonates, volcanics, cherts, and iron formations on several continents. His current research focuses on Precambrian spherule layers that represent distal ejecta from large asteroid impacts.



Kevin G. Taylor is a professor of sediment geochemistry and diagenesis at Manchester Metropolitan University, UK, where he has been based for 15 years; prior to this, he was a postdoctoral researcher at the University of Manchester. In his research, he applies electron-beam, mineralogical, and chemical analysis to the understanding of early diagenetic mineral precipitation in organic-rich mudstones and hydrocarbon reservoir–analogue sandstones; he has also carried out sediment–porewater studies in modern natural and contaminated sediment systems. A particular focus of his research has been on iron-rich sediments and rocks, including Mesozoic ironstones, iron minerals in urban geosystems, and modern tropical carbonate systems impacted by terrigenous sediment.



Alexis S. Templeton is an assistant professor of microbial geochemistry in the Department of Geological Sciences at the University of Colorado at Boulder. She is interested in understanding how microbial organisms conserve energy released during water–rock reactions in subsurface environments and in defining the structure and reactivity of biomineralization products. Her work involves the integration of synchrotron-based X-ray spectroscopy, aqueous geochemistry, isotope geochemistry, and environmental microbiology to unravel biological and abiotic pathways for the transformation of Fe, Mn, and S in modern systems.