J-PARC Workshop 2024

Deuterium Science Entering an Advanced Phase

October 18th, 2024
The Library Hall, Ibaraki University, Mito, Japan

Program

Sponsored by J-PARC Center

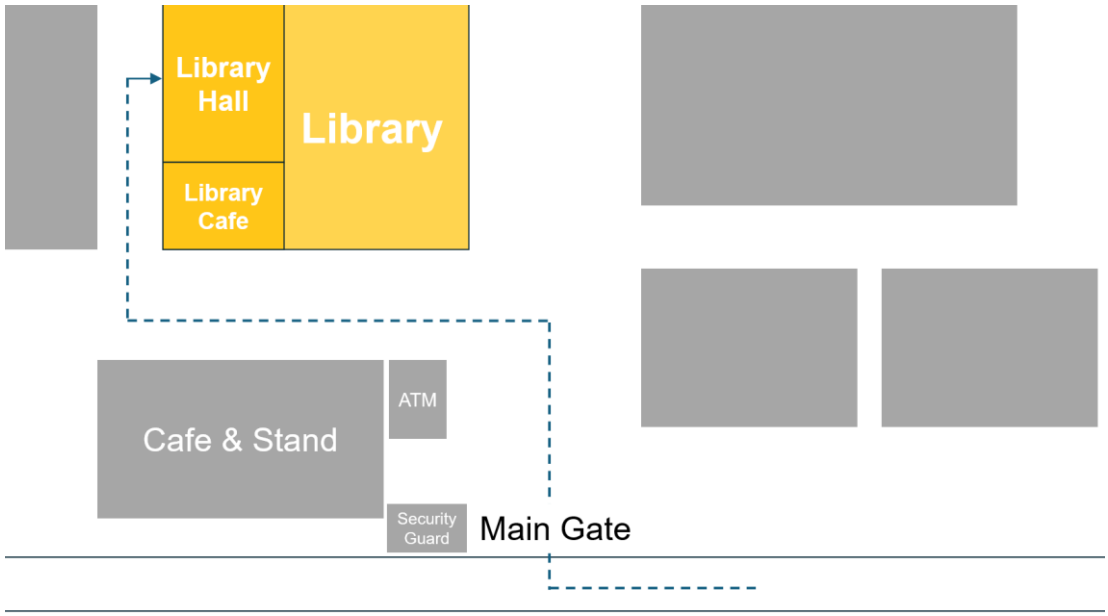
Co-sponsored by CROSS, J-PARC MLF Users Society, Institute for Integrated Radiation and Nuclear Science, Kyoto University

In cooperation with Research and Education Center for Atomic Sciences, Ibaraki University, and The Japanese Society for Process Chemistry

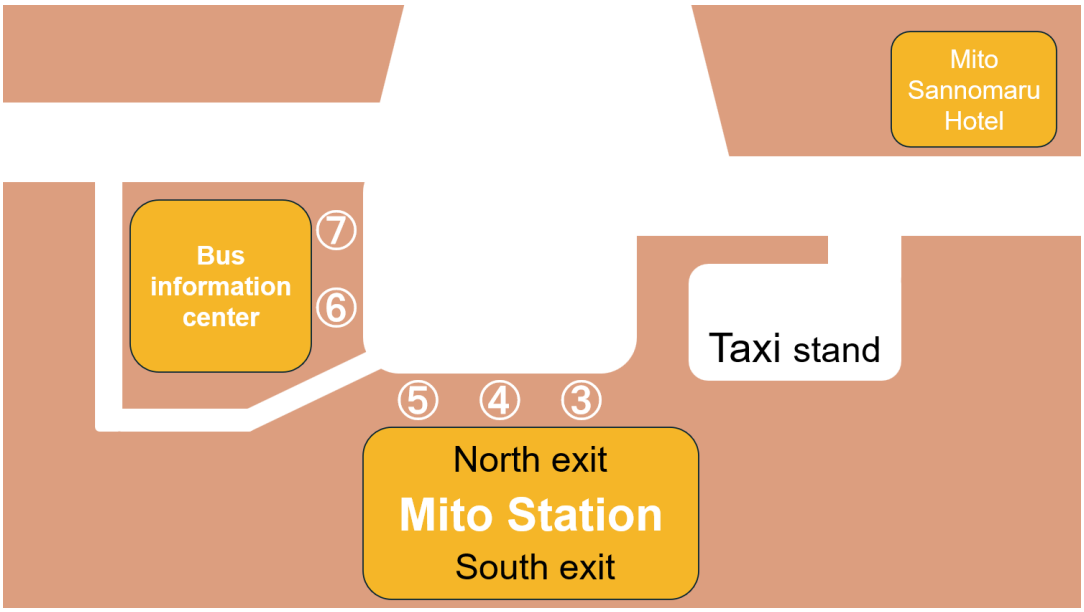
Programme at a glance

October 18th, 2024	
8:30-	Reception
9:00-9:15	Opening
9:15-10:00	Plenary Session 1
10:00-10:20	Group Photo & Coffee Break
10:20-11:20	Science Session 1 _Biology
11:20-12:20	Lunch & Poster session (core time)
12:20-13:05	Plenary Session 2
13:20-15:20	Science Session 2 _Featured Research from Overseas
15:20-15:40	Coffee Break
15:40-16:00	Science Session_3_Industry (Japanese Session)
16:10-17:10	Science Session_4_Chemistry
17:15-17:45	Free Discussion
17:45-17:55	Closing

Venue: The Library Hall, Mito Campus, Ibaraki University



Access (about 25 min): Take the bus bound for Ibadai (Ibaraki University) from JR Mito station (north gate) bus terminal 7 and get off at "Ibadai-Mae".



Banquet: The banquet will start at 19:00 at MITO KEISEI HOTEL (https://www.mito-keiseihotel.com).

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Program

9:00–9:10 Opening & Introduction of J-PARC MLF

T. Otomo (KEK/J-PARC Center)

9:10–9:15 Introduction of Quantum Beam Science at Ibaraki University

S. Mori (Ibaraki Univ.)

Plenary Session_1（Zoom）

Chair：M. Sugiyama (Kyoto univ.)

9:15–10:00 State of the art of biological deuteration at the Institute Laue-Langevin Frank Gabel (ILL)

Group Photo & Coffee Break (10:00-10:20)

Science Session_1 (Biology)

Chair：T. Ikegami (Yokohama city univ.)

10:20–10:50 Exploiting deuteration in BioSANS Anne Materl (ILL)

10:50–11:20 Protein deuteration in the deuteration laboratory of J-PARC MLF

T. Oda (J-PARC Center)

Lunch (11:20-12:20) & Poster session (core time)

Poster-01: Yao Chen (ISIS Facility), Deuteration Unveils the Adsorption and Self-Assembly Mechanisms of Polymeric Surfactants

Poster-02: Kazuhiro Akutsu, Misaki Ueda (CROSS), Chemical Deuteration Activities at CROSS Laboratory

Poster-03: Chie Shibazaki (J-PARC), Chemical Deuteration of Mono- and Di-Amino Acids for the Development of New Biopolymer Materials

Plenary Session_2（Zoom）

Chair：H. Sajiki (Gifu Pharm. Univ.)

12:20–13:05 Chasing the miracles of Science with Hydrogen Isotopes

Volker Derdau (Sanofi Germany)

Science Session_2 (Featured research from overseas**)**

Chair：M. Tachikawa (Yokohama city univ.)

13:20−14:00 Synthetic Biology and Flow Chemistry- Newly funded activities at the National Deuteration Facility for expanding the range of labelled molecules and their applications

T. A. Darwish (ANSTO-NDF)

14:00−14:40 ISIS Deuteration Facility and its influencing to ISIS neutron science Peixun Li (ISIS Deuteration Facility)

14:40−15:20 Catalytic isotopic labeling Wu Li (Wuhan Univ.)

Coffee Break (15:20-15:40)

Science Session_3 (Industry (Japanese Session))

Chair：K. Akutsu (CROSS)

15:40−15:45 Introduction of Industry session K. Akutsu (CROSS)

15:45−16:00 Heavy water recycling using waste power generation

M. Yoshida (Asahi Pretec)

Science Session_4_Chemistry

Chair：H. Sugiyama (CROSS)

16:10−16:40 A PIMD study of the hydrogen sulfate-formate proton-bound dimer

T. Udagawa (Gifu university)

16:40−17:10 A comparison from the perspectives of coordination structures, interfaces, and bulk extraction phases: fluorous and organic extraction systems

Y. Ueda (JAEA)

Recent activities of D-labeling in Japan

Chair：M. Adachi (QST)

17:15−17:45 Recent activities of D-labeling

T. Ikawa, H. Sajiki (Gifu Pharm. Univ.)

Summary

17:45−17:55 Summary talk & Closing M. Shibayama (CROSS)

Dinner (19:00-, @Mito Keisei Hotel)

O-2024-01

State of the art of biological deuteration at the Institute Laue-Langevin

Frank Gabel

Institut Laue-Langevin (ILL)

Neutron scattering is a unique technique allowing to access the structure and dynamics of matter at a molecular level, in complement to a multitude of other techniques, including optical spectroscopy, X-ray and electron scattering. It is particularly useful for biological samples due to the difference of scattering properties between hydrogen and its isotope deuterium, which allows to label selectively parts of complex molecular systems and focus on their respective structure (or dynamics), while masking (or diminishing) the signal from the remainder of the system.

I will present recent biological deuteration activities and examples from the ILL deuteration (D-) and lipid (L-) laboratories which are now grouped together in the newly founded BDCS group (“Biology, deuteration, chemistry and soft matter”) [1, 2]. Examples will include global deuteration strategies such as per-deuteration and match-out deuteration of biomolecules, including proteins and lipids, as well as more specific deuteration approaches such as segmental labelling and amino-acid labelling.

The benefit of the different labelling approaches will be illustrated by the specific structural and dynamic information that can be obtained by the associated neutron experiments.

References

[1] https://www.ill.eu/users/support-labs-infrastructure/deuteration-laboratory

[2] https://www.ill.eu/users/scientific-groups/soft-matter-science-and-support/deuterated-lipids-at-the-ill

Biographical Information

Frank Gabel has studied physics at Karlsruhe Technical University (now KIT) in Germany, and Joseph Fourier University (now UGA), Grenoble, France, from 1995 to 2000. He carried out a PhD thesis at the Institut de Biologie Structurale (IBS) from 2000-2003 under the supervision of Dr. Giuseppe Zaccaï and Dr. Martin Weik on protein dynamics studied by incoherent neutron scattering. From 2004-2006 he joined Dr. Michael Sattler's NMR group at EMBL Heidelberg, Germany, as a postdoc developing combined approaches of small angle scattering (SAXS/SANS) and NMR for structural studies of biomacromolecular complexes in solution. In 2006 he was recruited as a staff scientist at IBS Grenoble to promote the use of small angle scattering and integrative structural biology approaches within the Grenoble PSB (Partnership for Structural Biology). Since 2010, Frank Gabel has been working as a project leader in Dr. Bruno Franzetti's ELMA (Extremophiles and Large Molecular Assemblies) group at IBS. Since December 1st 2023 Frank Gabel has been appointed as head of the newly created "Biology, Deuteration, Chemistry and Soft Matter" (BDCS) group at the Institut Laue-Langevin (ILL), Grenoble. The group integrates staff from the deuteration, lipid and chemistry laboratories, as well as from the ILL laboratories of the PSCM (Partnership of Soft Condensed Matter) and from PSB. The tasks of the group include carrying out neutron-related developments and research in the fields of biology, deuteration, chemistry and soft matter, operating the laboratory user programs, strengthening interactions with the ILL user community and instrument groups, as well as promoting scientific collaborations within PSB and PSCM.

O-2024-02

Protein deuteration in the deuteration laboratory of J-PARC MLF

Anne Materl

Institut Laue-Langevin (ILL)

Deuteration is a required tool for several structural biology techniques. Amongst them, SANS takes advantage of this labelling to reveal the position of each partners in a macromolecular complexes. Through a few examples, I will show how deuteration can be designed, and combined with adequate sample environment, to get the most out of BioSANS measurement.

Biographical Information

Anne Martel did her PhD at the European Synchrotron Radiation Facility (Grenoble, FRANCE) and graduated in 2008. Then she worked as beamline scientist on the BioSAXS beamline of the SLAC Synchrotron Radiation Laboratory (Menlo Park, California) and as a post-doc on microfluidics at the Institut de Biology Structurale (Grenoble, FRANCE) before joining the Institut Laue Langevin as SANS instrument responsible in 2012.

O-2024-03

Protein deuteration in the deuteration laboratory of J-PARC MLF

Takashi Oda

J-PARC Center

Deuteration of samples is an important for structural analysis of biological macromolecules (proteins, nucleic acids) using neutron crystallography and small-angle neutron scattering (SANS). In the neutron crystallography, the use of fully deuterated proteins allows structural analysis with crystals smaller size than hydrogen proteins. In SANS, scattering from specific subunits or specific regions in protein complexes can be selectively observed by adjusting the deuteration rate of proteins and the concentration of heavy water in the solvent. For example, while a 75% deuterated protein is matched out in a 100% deuterated water solvent, light hydrogen proteins are not matched out under this condition, making it possible to specifically observe the scattering of light hydrogen proteins. Since the preparation of deuterated proteins requires a great deal of labor, we are developing a deuteration laboratory in J-PARC MLF and providing support for the preparation of deuterated samples for neutron users. In this session, we will talk the current state of deuteration laboratories and our research.

Biographical Information

Takashi Oda is a researcher of the J-PARC center of Japan Atomic Energy Agency in Tokai, Japan. He obtained his PhD degree from Yokohama City University (2010). He is a member of the deuteration laboratory in J-PARC MLF. He is interested in structure and function of intrinsically disordered proteins (IDPs), developing methodologies for structural and dynamical analysis of IDPs and deuterium-labelling of biological macromolecules. He is a member of the Japanese Society for Neutron Science, Protein science society of Japan, Japan Society for Bioscience, Biotechnology, and agrochemistry, the Biophysical Society of Japan.

O-2024-04

Chasing the miracles of Science with Hydrogen Isotopes

Volker Derdau

Sanofi Germany, R&D, Integrated Drug Discovery, Isotope Chemistry

One of the major motivations in pharma industry is to improve the life of patients using efficient drug treatments. Due to the complexity of biological processes in living creatures the number of scientific tools and experiments required to understand the nature of a disease is very high. One of these tools is the use of isotopically labeled compounds.[1] They contribute significantly to the deeper understanding of metabolism and the improved safety profile of drugs. While applications such as classical radioactive immune assays or nuclear magnetic resonance (NMR) structure elucidations lost their attractiveness due to other more accurate, simpler or faster methods, uses of isotopically labeled compounds have increased.

In recent years C-H functionalization of complex molecules has become a strong tool in lead optimization of bioactive molecules in the life science industry. To achieve this at the latest possible serial step is a convenient way to increase speed and to decrease costs and resources in drug discovery research. This concept is also followed in the Hydrogen Isotope Exchange (HIE[2]) reaction. The HIE reaction can be considered as the most fundamental of all C-H functionalization's as the proton is just substituted by its isotopes. In the lecture several concepts of HIE will be discussed with recent examples from the group and an outlook will be given how this isotope chemistry knowledge on CH-functionalization can foster lead optimization in drug discovery.

References

1. a) J. Atzrodt, V. Derdau, W.J. Kerr, M. Reid, *Angew. Chem. Int. Ed.* **2018**, 57, 1758-1784; b) V. Derdau, C. S. Elmore, T. Hartung, B. McKillican, T. Mejuch, C. Rosenbaum, C. Wiebe, Christine, *Angew. Chem. Int. Ed.* **2023**, e202306019.

2. a) J. Atzrodt, V. Derdau, M. Reid, W. J. Kerr *Angew. Chem. Int. Ed.* **2018**, *57*, 3022–3047; b) J. Atzrodt, V. Derdau, T. Fey, J. Zimmermann *Angew. Chem. Int. Ed.* **2007**, *46*, 7744–7765; c) Voges, R.; Heys, J. R.; Moenius, T. Preparation of Compounds Labeled with Tritium and Carbon-14; John Wiley & Sons, **2009**; d) S. Kopf, F. Bourriquen, W. Li, H. Neumann, K. Junge, M. Beller *Chem. Rev.* **2022**, 122, 6, 6634–6718; e) Q.-K. Kang, H. Shi, *Synlett* **2022**, 33, 329-338; f) V. Derdau **2022** in Handbook of CH-Functionalization (ISBN 9783527834242. Ed. A. Maiti).

Biographical Information

Volker Derdau studied chemistry in Münster and Braunschweig (Germany) and obtained his PhD in 1999 with Prof. Sabine Laschat. He went for a one-year Deutscher Akademischer Austauschdienst (DAAD) funded Post-Doc in Prof. Victor Snieckus group (Kingston, Canada) before he started at Aventis Pharma Germany as laboratory head in Chemical Development. Today he is Department Head and Senior Distinguished Scientist in the Integrated Drug Discovery platform (Sanofi, Frankfurt) responsible for Isotope Chemistry, Center of Excellence. He is Editor-in-Chief of the Journal of Labeled Compounds and Radiopharmaceuticals (Wiley) and author of more than 80 publications, three book chapters and owner of eight patents. He has co-organized some International Isotope Society (IIS)- European Division (ED) workshops, the global IIS-conference in Heidelberg 2012 and is member of the IIS Board of Trustees (BoT) since 2012. Since 2017 he is elected president of the IIS-ED chapter and became global IIS president in 2021 and 2023. Furthermore, he is interested in teaching and was lecturer at the University of Applied Sciences in Darmstadt (2012-2018) and the Provadis Academy Frankfurt/Germany (2021 – present). He was recipient of the IIS-ED prize in 2007 and of the European Isotope Science Award in 2022. In his free time, he acts as judo referee on national level or is working as consultant.

O-2024-05

Synthetic Biology for expanding the range of isotopically labelled molecules produced at the National Deuteration Facility

Tamim Darwish

the National Deuteration Facility (NDF)

The diversity of applications of labelled molecules mandates continuous development of new production methods that are efficient, sustainable and economically viable. Synthetic biology is one of these methods that can be used for synthesizing deuterated molecules that are difficult to produce by chemical means alone, or when other stable isotope labelling (^{13}C and ^{15}N) is required. To incorporate ^{13}C and ^{15}N stable isotopes in the backbone of organic molecules, synthetic biology becomes the only efficient and cost-effective method to produce such type of molecules. The expansion of biosynthetic capability of the National Deuteration Facility at ANSTO has provided a greater range of molecules for neutron, NMR, MS and other research techniques. Deuterated molecules currently available via genetic modification of Saccharomyces cerevisiae include: cholesterol, campesterol, desmosterol, squalene (the precursor to sterols and other lipids), which will be discussed in the presentation.[1] Labelled polyunsaturated fatty acids such as DHA (docosahexaenoic acid) are produced by single-cell marine Thraustochytrids. Deuterated cholesterol and other sterols have been used in neutron scattering investigations of SARS-CoV-2 infection and mRNA vaccine development.[2,3] Metabolic studies using deuterated and ^{13}C labelled DHA[4] or deuterated cholesterol[5] also employ isotope-sensitive instruments such as NanoSIMS for detecting the fate of molecules in various body tissues. Deuterated squalene showed enhanced oxidative stability over its protiated analogue.[6]

References

1. Recsei, C., et al. *Organic and Biomolecular Chemistry* **2023**, 21, 6537-6548.
2. Sebastiani, F., et al. (2021). *ACS Nano* **2021**, 15, 6709-6722.
3. Santamaria, A., et al. *Journal of the American Chemical Society* **2022**, 144, 2968-2979.
4. Chen, K., et al. *Journal of lipid research* **2022**, 63, 100290.
5. Ferrari et al. *Science* **2023**, 382, eadf0966.
6. Möller, J., et al. *RSC Advances* **2024**, 14, 26002-26006.

Biographical Information

Tamim is the Director of the National Deuteration Facility (NDF) at ANSTO. He is an Honorary Professor of the University of Canberra. He manages and oversees the operations of both the chemical and biological deuteration laboratories that make up the National Deuteration Facility. He manages the development of new capabilities, scientific activities, research outcomes and user/industry engagement for the facility.

O-2024-06

ISIS Deuteration Facility and its influencing to ISIS neutron science

Peixun Li

ISIS Pulsed Neutron and Muon Source, Science and Technology Facilities Council, UKRI

The ISIS Deuteration Facility significantly enhances the capabilities of ISIS neutron science, providing essential deuterated materials and expertise for advanced neutron scattering experiments. The facility supports a wide range of research areas, including materials science, biology, and chemistry, by enabling precise measurements of molecular structures and dynamics. By offering custom synthesis of deuterated compounds, the facility ensures that researchers have access to tailored materials that meet their specific experimental needs. This capability is particularly important for studying complex systems where contrast variation is required to discern structural details. The Deuteration Facility not only broadens the scope of experiments that can be performed at ISIS but also improves the accuracy and reliability of the results. Through collaborations with both academic and industrial partners, the facility fosters innovation and drives forward the frontiers of neutron science. Overall, the Deuteration Facility is a key infrastructure of ISIS, enhancing the research output and scientific impact of neutron scattering studies.

Biographical Information

Peixun Li is the scientific lead for the ISIS deuteration capability, specializing in the study of surfactant, polymer, and lipid mixtures at interfaces using neutron scattering techniques. He has authored over 150 peer-reviewed papers in materials chemistry, soft matter, and neutron scattering. His primary focus is on chemical deuteration, widely applied across fields such as organic chemistry, organometallic chemistry, pharmacology, and soft matter.

Dr. Li has established extensive collaborations with the neutron science community in both academia and industry. He has led or participated in over 40 projects related to chemical deuteration, playing a key role in advancing research in materials chemistry and soft matter.

O-2024-07

Catalytic isotopic labeling

Wu Li

Wuhan University

Deuterium labeling has widespread applications in medicinal chemistry and material science. For example, deuterated drugs e.g. Austedo, Donafenib, Sotyktu and VV116 have been approved, respectively. Because of these applications, the development of methodologies for the synthesis of deuterated compounds with high deuterium incorporation is highly desirable. However, the development of methodologies for large-scale deuterium labelling and reductive deuteration of aromatic hydrocarbons, have been rarely achieved. In the context, we have developed an easily scalable deuteration of (hetero)arenes via H/D exchange and a general electrocatalytic method for the reductive deuteration (hetero)arenes (see the figures below).[1-2] In addition, some other deuterium labeling methods have also been developed, recently. [3-5] The developed protocols have been successfully applied to the synthesis of deuterium incorporated drug molecules.

References

[1] W. Li et al, *Nature Chem.* **2022**, 14, 334–341.

[2] W. Li* et al, *Nature* **2024**, doi: 10.1038/s41586-024-07989-7.

[3] W. Li* et al, *Green Synth. Catal*. **2024**, doi: org/10.1016/j.gresc.2024.06.003.

[4] W. Li* et al, *Chin. J. Chem*. **2024**, 42, 1145–1156.

[5] W. Li* et al, *Chin. J. Chem*. **2024**, doi: 10.1002/cjoc.202300644.

Biographical Information

Prof. Wu Li was born in 1986 in Anhui province, China. He received his Ph.D. degree in 2015 from Wuhan University under the supervision of Prof. Aiwen Lei. In 2016–2021, he worked as a postdoctoral researcher with Prof. Matthias Beller in Leibniz Institute for Catalysis (Germany). In 2022, he got a professor position in College of Chemistry and Molecular Sciences (CCMS) of Wuhan University. His current research interest is catalytic isotopic labeling.

O-2024-09

A PIMD study of the hydrogen sulfate-formate proton-bound dimer

Taro Udagawa
Gifu University

Hydrogen bonds are basic but extremely important structural motifs in chemistry, physics, and biochemistry. The ionic hydrogen bond is one of the special classes of hydrogen bonds due to its strong interaction. Proton-bound dimer (PBD) compounds, where a proton is shared by two Brønsted conjugate bases, are a kind of ionic hydrogen bond compound. The PBD compound of hydrogen sulfate and formate has low-barrier hydrogen bonds; thus, nuclear quantum effects (NQEs) should be considered in theoretical analysis of this PBD compound. The structure of the PBD compound of hydrogen sulfate and formate has been studied using the path integral molecular dynamics method, which can incorporate NQEs. This study addressed the following question: “Is the shared proton localized on either an anion or located around the center of two anions?”. We have elucidated that although the shared proton did not completely overcome the transition state for the proton shuttle, the shared proton was distributed in the region beyond the transition state due to the NQEs.

Biographical Information

Taro Udagawa is an associate professor at Gifu University, Japan. He earned his PhD from Yokohama City University in 2008 under the supervision of Prof. Masanori Tachikawa. In April 2008, he began his career as an assistant professor at Gifu University and he was promoted to associate professor in 2023. His research interests focus on H/D isotope effects and nuclear quantum effects, and he works on developing new quantum mechanical methods to analyze these phenomena. He is a member of the Chemical Society of Japan, the Japan Society of Theoretical Chemistry, the Japan Society of Molecular Science, and the Society of Computer Chemistry, Japan.

O-2024-10

A comparison from the perspectives of coordination structures, interfaces, and bulk extraction phases: fluorous and organic extraction systems

Yuki Ueda

Materials Sciences Research Center, Japan Atomic Energy Agency

Microscopic structures in liquid–liquid extraction, such as structuration between extractants in bulk organic phases and at interfaces, can influence macroscopic phenomena, such as the distribution behavior of solutes and phase separation of the organic phase. We correlated the macroscopic behavior of the extraction of Zr(IV) ions from HNO_3 solutions with microscopic structural informatio in order to understand at the molecular level the key factors contributing to the higher metal ion extraction performance in the fluorous phosphate in perfluorohexane as compared to the analogous organic phosphate (THP) in n-hexane. Extended X-ray absorption fine structure (EXAFS), neutron reflectometry (NR), and small-angle neutron scattering revealed the local coordination structure around the Zr(IV) ion, the accumulation of extractant molecules at the interface, and the structuration of extractant molecules in the bulk extraction phase, respectively. In this study, deuteration of THP was performed to ensure a difference in neutron scattering length density between extractant and diluent in the NR experiment. This presentation will mainly report on the relationship between microscopic structural information obtained from EXAFS, NR, and SANS and macroscopic extraction behavior, and discuss the factors that give rise to differences between fluorous and organic extraction systems.

Biographical Information

Dr. Ueda obtained his Ph. D (Engineering) from Saga University, Japan, in 2017 in the field of separation engineering. He worked as a postdoc researcher at JAEA (2017–2020) and started to research of design and synthesis of extraction reagents. He then became a permanent staff of JAEA in 2020, and is currently a researcher of the institute. His recent research mainly focuses on the development of separation techniques for radionuclides using liquid–liquid extraction methods. The main research fields are nuclear and metal separation chemistry.

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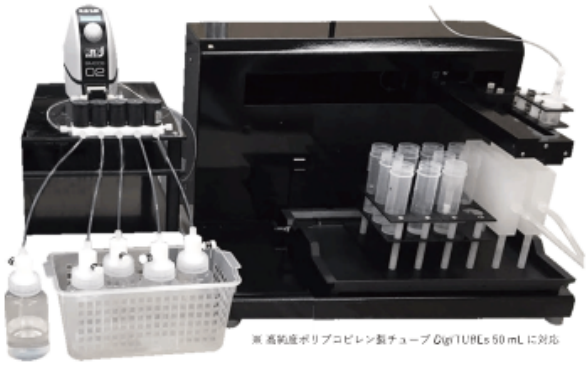

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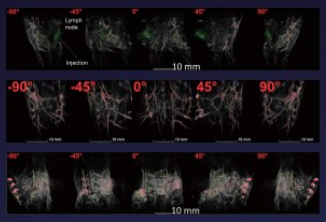

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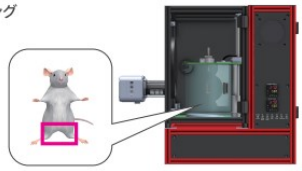
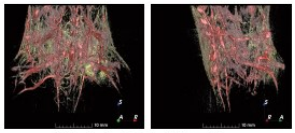
・解剖基準点（皮膚、血管系、内臓）
・光吸収バイオセンサー



同時に同一箇所を測定 励起波長掃引による分子イメージングも可能

マウスの血管イメージ



様々な角度から観察可能！

3Dカラーイメージ動画を
弊社HPにて掲載中▶

用途・アプリケーション

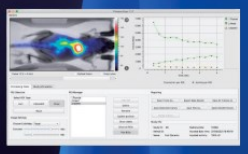
●光音響トモグラフィー（PAT）
●蛍光分子トモグラフィー（FMT）
●選択的な腹部、骨盤、胸部、皮膚のトモグラフィー
●血管および臓器 のイメージング
●色素やナノ粒子などの光プローブを用いたイメージング
●遠赤外および近赤外蛍光プローブによるイメージング

BIOEMTECH

前臨床試験用途に活躍中！

ベンチトップPET診断装置（マウス）
eyeシリーズ

■ 2 次元画像をリアルタイムに測定
■ シンプルな操作で in vivo の反応を観察するのに最適
■ 従来の 3 次元観察装置よりも簡単でより早く
ドラッグデリバリーなどの観察が可能



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